THE FINAL MASQUERADE: A MOLECULAR-BASED APPROACH TO THE IDENTIFICATION OF RESINOUS PLANT EXUDATES IN ROMAN MORTUARY CONTEXTS IN BRITAIN AND EVALUATION OF THEIR SIGNIFICANCE

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Rhea Catharine BRETTELL

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To every man upon this earth
Death cometh soon or late.
For how should man die better,
Than facing fearful odds,
For the ashes of his father
And the temple of his gods?

Horatius, The Lays of Ancient Rome
Thomas Babington Macaulay 1881

Rhea Catharine BRETTELL

The final masquerade: a molecular-based approach to the identification of resinous plant exudates in Roman mortuary contexts in Britain and evaluation of their significance

Key words: molecular analysis; resinous plant exudates; GC-MS; mortuary rites; body treatment; materiality; Roman Britain

Abstract

This study provides chemical confirmation for the use of resinous plant exudates in mortuary contexts in Roman Britain. Analysis of amorphous masses, adhering residues and grave deposits using gas chromatography-mass spectrometry has revealed terpenoid biomarkers in sixteen inhumation and two cremation burials. The natural products characterized include European Pinaceae (conifer) resins, *Pistacia* spp. (mastic/terebinth) resins from the Mediterranean or the Levant and *Boswellia* spp. (frankincense) gum-resins from southern Arabia or eastern Africa. In addition, traces of a balsamic resin, probably *Liquidambar orientalis,* have been identified. A correlation between the use of these exotic exudates and interment in substantial, often multiple, containers with high-quality textiles and grave goods was observed.

Theoretical consideration of this imported rite illuminates the multiplicity of roles played by resins/gum-resins in the mortuary sphere. The material properties of these highly scented substances speak to the biological reality of the decomposing body and to the socially constructed identity of the individual. On a practical level, they acted as temporary preservatives and masked the odour of decay. As social signifiers, they denoted the status of the deceased and promoted remembrance through conspicuous consumption and sensory impact. Encoded with ritual meaning, they purified the body and facilitated the final rite of passage to the afterlife. The recovery of these resinous traces provides us with new insights into the treatment of the body in the Roman period and establishes fresh links between the remote province of *Britannia* and the remainder of the Empire.

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Introduction: questions and aims

"Here I lament for you with funeral gifts and eulogy...no sweeter scent comes from Sicilian saffron, nor rich Sabaeans's special cinnamon picked just [for you]...nor from the Arabs' crop, their fragrant frankincense". Statius Silvae 5.3:53-59 no date (nd)

1.1 Background

During the past thirty years improved excavation techniques and the publication of detailed cemetery reports have provided evidence for considerable complexity in mortuary rites in Britain during the mid-late Roman period (2nd- early 5th centuries AD) (e.g. Barber and Bowsher 2000; Booth *et al.* 2010; Davies *et al.* 2002; Farwell and Molleson 1993; Ottaway *et al.* 2012). This facet of Roman rule has, however, remained somewhat neglected as a research area (Esmonde Cleary 1992; Morris 1992; Reece 1982; Thomas 1981: 228-230), particularly in relation to current theoretical approaches regarding ritual action within the mortuary sphere (Pearce 2000). Indeed, "*Roman death as a subject...*[has rarely been]*...viewed as an integrated topic*" (Hope 2009: 3) and so both practical and ritual aspects of the treatment of the dead during the Roman period have received less attention than is the case for other archaeological/historical eras (e.g. prehistory, Anglo-Saxon) (Pearce 2000).

One aspect of Roman practice that warrants attention is the reported presence of resinous substances in mortuary contexts from Britain. Fuelled by the brief insights provided by classical authors (Hope 2009: 65-85), for centuries there has been unsubstantiated speculation regarding the presence of exotic plant exudates in both cremation (Gage 1834, 1836; Waugh 1962) and inhumation burials (Dunkin 1844: 91-97; Morris 1986; Taylor 1993; Webster 1950; Woodward 1993: 216-239). Moreover, during the 1970s, a specific class of late Roman burial rite was proposed based on an apparent correlation between substantial, often multi-element, burial containers (stone sarcophagi and/or lead-lined coffins) and enshrouded bodies encased in plaster (Ramm 1971). The potential for resinous substances to comprise part of this 'package' was also mooted as the additional level of investment, which took the form of increased protection of the cadaver, appeared to be aimed at some degree of body preservation (Sparey Green 1977).

The significance of this practice has been extensively debated. Inferential links with the doctrine of resurrection, in conjunction with reports of plaster in late Roman burial contexts in northern Africa and the Rhineland, led to an association with the spread of Christianity (Ramm 1971; Sparey Green 1977, 2003). The likelihood of any such relationship has, however, been disputed and, as initially posited by Ramm (1971), its potential as a signifier of social status has been proffered as an alternative explanation (Philpott 1991: 92; Toller 1977: 2). Associations with other religious cults and gender correlates have, likewise, been advocated (Chioffi 1998: 24-28; Morris 1986; Philpott 1991: 92; Toynbee 1996: 42; Woodward 1993: 236-237). Despite widespread interest in the possible use of plant exudates (Pearce 2013), no chemical analysis has previously been carried out in Britain either to confirm the presence of resins or to identify their botanical origins. Thus, interpretations of this body treatment have mentioned likely sources and speculated on the ritual meaning of this rite in an absence of definitive evidence. Along the way, a mythology has developed through repetition of earlier suppositions.

A range of instrumental techniques can be applied to the identification of natural products in the archaeological record (Evershed 2008a; Pollard and Heron 2008: 235-269). This includes the characterisation of plant exudates, the sticky scented substances secreted when the bark of certain trees or shrubs are 'wounded' (Langenheim 2003: 23-26). Based on their chemical composition, these can be grouped into gums (water-soluble polysaccharides), resins (water-insoluble terpenoids) and gum-resins (variable amounts of both fractions). The material properties of these substances have resulted in their widespread anthropogenic exploitation as adhesives, protective coatings, varnishes and illuminants with the more fragrant varieties employed as components of perfumes, unguents, medicines, embalming mixtures and as incense (Mills and White 1977; Pollard *et al.* 2007: 153-156; Serpico and White 2000). Fortunately, their use in antiquity can often still be traced as the higher molecular mass (HMM) terpenic compounds in the resin fraction are relatively resistant to decay and so survive well in the archaeological record (Pollard and Heron 2008: 235-269). Chemical analysis of organic residues, principally through the use of gas chromatography-mass spectrometry (GC-MS) and the biomarker approach, can reveal such evidence and effectively render the invisible, visible (Evershed 2008a). This technique has added considerably to our knowledge concerning the past exploitation of these resources and illuminated questions regarding technological developments, trade and tribute relations and ritual activities (Modugno and Ribechini 2009; Serpico 2000).

With regards to the use of plant exudates, archaeological mortuary contexts have proved a particularly fruitful area of inquiry. Much of this research has focussed on Ancient Egyptian mummification processes (e.g. Buckley *et al.* 1999; Charrié-Duhaut *et al.* 2007; Colombini *et al.* 2000; Hamm *et al.* 2004; Koller *et al.* 1998; Serpico and White 2000) and their continuance during the Greco-Roman Period (e.g. Buckley and Evershed 2001; Corcoran and Svoboda 2010; Maurer *et al.* 2002; Proefke and Rinehart 1992; Tchapla *et al.* 2004). These studies have demonstrated that, even in complex mixtures such as embalming materials, suites of marker compounds permit the identification of aromatic plant products and that resinous substances played a key role in soft-tissue preservation. They have also revealed that a modified form of this body treatment continued into the Roman period in Egypt with a significant increase in the use of imported conifer resins (Buckley and Evershed 2001; Corcoran and Svoboda 2010).

Such findings prompted the analysis of visible residues from exceptional mortuary contexts elsewhere in the Roman Empire. Biomarkers denoting the utilization of both diterpenoid conifer products (principally Pinaceae resins) and triterpenoid (*Pistacia* spp.; *Boswellia* spp.) exudates from late Roman period inhumations in Italy (Ascenzi *et al.* 1996; Bruni and Guglielmi 2005; Devièse *et al.* 2010), Greece (Papageorgopoulou *et al.* 2009), France (Devièse 2008: 115-131) and the Rhineland (Reifarth 2013: 91-114) have been identified. Thus, it appears that resinous substances were indeed incorporated into burials outside Egypt. These finds display a number of commonalities which closely resemble the class of late Roman burial proposed by Ramm (1971) with the deceased generally interred in stone sarcophagi and/or lead-lined coffins and accompanied by high quality grave goods (Ascenzi *et al.* 1996; Bruni and Guglielmi 2005). Most appear to have been wrapped in a shroud with several encased in plaster or surrounded by wood shavings (Reifarth 2009). Evidence for garments of damask silk, shellfish ('Tyrian') purple-dyed wool and spun gold threads has also been recovered (Devièse 2008: 181-182; Papageorgopoulou *et al.* 2009; Reifarth 2013: 47-90).

But what of the remote province of *Britannia*? Would there have been a demand at the far northern edge of the Empire for these exotic resins and accompanying rites? The extent to which Roman culture permeated society in Britain has long been the subject of heated debate (Versluys 2014) as captured in the famous passage penned by Tacitus in around AD 98 (*Agricola 21* nd): "*and so they strayed into the enticements of vice*...[which]...*in their innocence they called 'civilisation', when in fact it was a part of their enslavement*". That novel mortuary practices were introduced is certainly well-attested as urned (or otherwise contained) cremation followed by inhumation in extra-mural burial grounds was adopted in contrast to the largely archaeologically-invisible body disposal methods of the pre-Roman Iron Age (Philpott 1991; Whimster 1981). In addition, increased evidence for the use of plaster (e.g. Barber and Bowsher 2000: 320-321; Philpott 1991: 90-96; Woodward 1993; Whytehead 1986) and, most recently, high quality textiles with gold thread and murex purple-dyed decoration in a number of inhumation burials (Walton Rogers 2002; Wild 2013) supports the supposition that more elaborate body treatments were part of the range of funerary choices available to individuals in Roman Britain.

In what form, if any, would evidence for ritual actions involving resinous substances survive? The majority of the previous finds from continental Europe derive from relatively well-preserved visible residues (e.g. in sarcophagi from Trier, Reifarth 2013: 185-430). In Britain, however, such samples are less likely to be encountered due to the absence of upstanding Roman period mortuary structures (e.g. mausolea, burial vaults or catacombs) combined with inclement environmental conditions, in particular the impact of water movement. Moreover, due to their amorphous nature and tendency to become increasingly friable over time resinous substances are often overlooked during excavation or by conventional analytical approaches (Evershed *et al.* 1997a). A key facet of the research was, therefore, not only to evaluate visible fragments and residues but also other samples with the potential to retain chemical information regarding the treatment of the body such as the comminuted debris from the base of the more substantial burial containers. This potentially informative material is often discarded in favour of the recovery of ecofacts and artefacts so this aspect of the project was viewed as an opportunity to highlight the level of information that can be obtained from analysis of such grave deposits. The sampling and retention of realistic amounts of suitable materials could then be promoted for inclusion at the planning stage of the excavation of burials of this nature.

With these provisos, archaeological mortuary contexts in Britain can be considered to be an highly appropriate context for such research. They are sufficiently defined in both space and time to enable a systematic study of extant examples of the proposed burial class and comparative materials to be undertaken. In addition, the recovery of biomarkers from these unprepossessing samples can be attributed to archaeological sources with some confidence as only one native species, *Pinus sylvestris*, readily produces a terpenoid-containing resin and then not in any significant abundance (Howes 1949: 109). As the natural distribution of this conifer is in northern, upland areas of Britain while issues of access and availability restricted sample acquisition to southern and/or lowland sites, the potential for contamination within the surrounding soil matrix was considered to be minimal. Thus, any chemical evidence for resinous exudates in Roman period burials of this nature offers a rare opportunity to discern the actions that created this archaeological mortuary record.

1.2 Research aim and thesis outline

These observations gave rise to a number of research questions:

1. were resinous plant exudates used in mortuary contexts in Roman Britain?
2. if so, which botanical sources were selected and where were their geographical places of origin?
3. how were the resins treated and applied and what was their purpose?
4. can a coherent burial class be defined based on the presence of resins and do commonalities exist with other material aspects of this rite?
5. how is this body treatment situated in relation to normative mortuary practices in Roman Britain and to burials from continental Europe and North Africa incorporating resinous substances?
6. can the chemical identification of resinous substances in conjunction with mortuary theory permit the social and ritual actions encapsulated in this rite to be deciphered?

The aim of this thesis is, therefore, to apply a molecular-based approach to identify the source(s), method of application and purpose of resins found in mortuary contexts in Roman Britain and to illuminate the nature of this rite through comparison with similar burials in Roman Europe. In order to address these issues, the research follows a bipartite approach combining chemical analysis of systematically selected samples from a range of mortuary contexts and the application of current mortuary theories to address previous, unsubstantiated speculation regarding a) the presence and botanical nature of resinous substances in Roman period burials in Britain and b) their purpose and relationship to ritual action within the wider context of the Roman Empire.

The two main hypotheses are that:

1. resinous substances were transported to Britain during the mid-late Roman period (2^{nd}-early 5^{th} centuries AD) and used in mortuary contexts;

2. these plant exudates, in conjunction with substantial burial containers, plaster body-casings and textiles, held a specific social and/or ritual meaning.

To test these premises, current theoretical approaches to the interpretation of the mortuary record are evaluated alongside information from primary sources regarding Roman responses to death (**Chapter 2**). The specifics, in terms of the treatment of the body, are then considered based on references in the classical literature and archaeological evidence from continental Europe (**Chapter 3**) with burial practices in Roman Britain reviewed in order to compile a list of burials of interest (**Chapter 4**). In **Chapter 5** the chemistry of resinous substances is addressed focussing on those botanical sources potentially available within the Roman Empire whose exploitation in ritual contexts has been attested or suggested. Information relating to the impact of natural environmental and anthropogenic degradation (e.g. heating) on these substances is also amassed.

The protocols devised for sample collection, preparation and instrumental analysis are detailed in **Chapter 6** and relevant issues evaluated (e.g. sample availability, selection of controls). Data was obtained using a range of appropriate complementary non-destructive (Fourier Transform infrared spectroscopy; Raman spectroscopy) and minimally-destructive (GC-MS) techniques with modern, botanically and geographically sourced reference materials analysed as comparatives and the purchase or biosynthesis of standards carried out in order to aid identification and supplement the spectrometric literature. The results are presented as a series of case studies, each detailing the archaeological context of the extant samples obtained from each region, site or individual burial (**Chapter 7**). In order to identify any commonalities, these findings are integrated within a multidisciplinary framework through consideration of osteological and palaeopathological data, body orientation, the form of burial container and the nature of any grave goods, textiles or other anthropogenic inclusions such as plaster body-casings. This aspect of the research benefited greatly from collaboration with other analysts (e.g. Jacqueline McKinley and

Rebecca Redfern, human remains; Eline Schotsmans, plaster; Penelope Walton Rogers, textiles), as referenced in the relevant case studies, and led to a number of publications (**Appendix 7.5**).

Chapter 8 provides a synthesis of the preceding research with the empirical data contextualised in terms of both the practical and theoretical aspects of Roman mortuary practices. The research questions are addressed in relation to the social, cultural and economic links between Britain and the rest of Empire and the various threads drawn together in order to interpret the meaning of this rite. In conclusion (**Chapter 9**), the hypotheses laid out above are discussed in order to bring to an end centuries of speculation regarding the presence and nature of resinous substances in Roman period burials in Britain, afford novel insights into the rationale behind this treatment of the dead and re-position ritual action in the province within the wider geographical and socio-political context of the late Roman Empire. The direction of future research is then considered.

Chapter 2
Roman mortuary rites: theory and practice

"The life of the dead is [situated] in the memory of the living".
Cicero, Phillipics 9:5 nd

2.1 Introduction

"Mortuary evidence is an extremely valuable archaeological resource, since it represents the direct and purposeful culmination of conscious behaviour, rather than its incidental residue" (O'Shea 1981: 39). Burial practices are essentially concerned with the disposal of the decaying human body and yet the momentous nature of death means that they also have the potential to reveal much about the lived social context (Shanks and Tilley 1982). One of the most prescribed and structured deposits encountered by archaeologists, the treatment of the dead is an highly significant act imbued with symbolic meaning so "special attention [should be paid] to the social aspects...revealed" (Hodder 1980: 161). The full potential of this repository of material evidence is all too often overlooked or inadequately addressed (Shanks and Tilley 1982; Morris 1992: 1-3) since moving beyond a simplistic discussion of things to read the underlying behavioural text presents a considerable challenge (Hodder 1986: 13). Approaches to our universal fate are both diverse and culturally situated (Metcalf and Huntington 1991). Nonetheless, as decades of research in the social sciences have shown, commonalities can be discerned and questions about the significance of specific mortuary rites in a given archaeological context investigated through reflection on the active role of materialities (Fahlander and Oestigaard 2008; Hertz 1960).

In terms of Roman burial practices, theoretical approaches of this nature have rarely been applied so neither the method nor meaning of processes involved in the preparation of the body has received much attention. Neglect of this aspect has drawn comment over the years (e.g. Esmonde Cleary 1992; Graham 2011; Reece 1982) but the potential contribution of the dead still lags behind a focus on political and economic power relations (Hope 2011). Indeed, research in general has suffered heavily from entrenched archaeological ideologies, particularly in English language publications, as characterised by the long-running debate over the relevance of the 'Romanisation' paradigm (Millett 1990; Pearce 2000; Versluys 2014). Thus, even the best reviews, in relation to Roman Britain, make limited use of mortuary evidence and rarely consider specifics concerning the treatment of the dead (e.g. Mattingly 2006; Potter and Johns 1992; Salway 1981; Southern 2013). A very significant trick is being missed. Roman mortuary contexts provide direct, constrained information generally related to a single event replete with materiality and meaning and whose taphonomic pathways can, to some degree, be predicted or experimentally determined.

To rectify this situation, Woolf (2014) has proposed that researchers should focus on small, well-defined, targets using all the available evidence (e.g. literary, epigraphic and archaeological) to consider the meaning of material aspects of culture and reveal associations. The decision was taken, therefore, to focus on one province of the Roman Empire, Britain, and one rarely investigated aspect of Roman mortuary rites, the use of resinous substances in the treatment of the body. To place this research in context, a review of recent developments in mortuary theory was undertaken (**2.2**) and our current understanding of Roman approaches to death and the afterlife assessed (**2.3**). A summary of the main points is provided in **2.4**.

2.2 Theorising mortuary practices

2.2.1 Dealing with death

"A time of change, transformation and transition" (Hope 2011: xix) not only for the deceased (physically and spiritually) but also for the survivors (from the immediate family and friends to the wider community), dealing with death entails a fundamental readjustment in terms of both individual and social relations (Hertz 1960). The significance of death means that humanity's responses are always "meaningful and expressive" (Metcalf and Huntington 1991: 24) but they have also been shown by ethnographers and other social scientists to be culturally-situated and highly varied. Despite differing material manifestations, a consistent underlying sequence of ritual action has been revealed in the conduct of this final rite of passage (Hertz 1960). The three stages identified comprise: the moment of separation, a period of transition and the final act of incorporation. Through this process, the successful transference of the ‘spirit’ of the deceased to the otherworld and the return of the survivors to their transformed lives within the re-balanced social order is achieved (van Gennep 1960).

These observations regarding the significant role of mortuary rites in manipulating and maintaining social structures led to the realisation that a body of theory was required to enhance the scope of archaeological interpretation (Parker Pearson 1982). As reviewed by Chapman and Randsborg (1981), early work by Ucko, Binford and Saxe sought to identify commonalities and regularities in burial rites in order to draw inferences about past social structures based on data patterning and ethnographic analogies. This was built on by Tainter (1975, 1978) using energy expenditure models and a more critical approach to social typologies. Modified by an understanding of the importance of context and the necessity to consider subversion and inversion, as expressed in the work of Hodder (1980, 1986), many of these ideas have stood the test of time. Certain mortuary practices do, indeed, seem to be consistently associated with aspects of actively negotiated social identity, for example the complexity of body treatment and nature of material contributions, with the quality rather than quantity of grave goods often key in signifying status (Parker Pearson 2003: 21-44).

Such studies served to reveal the central role of material culture in embodying and mediating relationships between the living and the dead (Hallam and Hockey 2001: 13-19). Material symbolism pervades all societies and forms an integral part of every cultural package (Pader 1980). The tangible remains of ritual action recovered from the archaeological mortuary record should, therefore, allow us to reconstruct associations within different symbolic systems (Parker Pearson 1982: 110). As with all forms of communication, however, the discourse of things is complex, fluid and subjective. Meaning lies in the ‘mind’s-eye’ of both the observed and the observer (Barrett 1991). Thus, the importance of situating archaeological mortuary data within its cultural context, in terms of the relationship between the society under consideration and its practices for disposal of the dead, is crucial (Chapman and Randsborg 1981). Fortunately, the repetitive nature of ritual action is often more bounded and constrained in the context of burial practices than in other social arenas enabling aspects of identity to be discerned (Parker Pearson 1982: 100-101).

2.2.2 Materialising meaning

This nexus between anthropological theory and archaeological thought has, in recent years, resulted in an increased interest in the materiality of death. Incorporating the concept of agency (the capacity of an entity to act within a given environment) within the context of materialities (the social significance of entities), this approach advocates an explicit focus on the active role played by material culture in constituting social relations (Fahlander and Oestigaard 2008). Its application has led archaeologists to seek alternative ways of thinking about their data and address new questions. Thus, a number of recent studies have demonstrated the need to “unite the social and biological aspects of death” (Graham 2011: 21) through consideration of the corporeality of the body, its transformations after death and the sensory impact of the decomposing cadaver in conjunction with social constructs such as identity (Stutz 2008). The importance of reflecting upon the role of material domains, including factors such as smell, as mitigators of loss and mediators of memory has also been highlighted (Hallam and Hockey 2001: 17-26; Graham 2011).

So, how can we hold meaningful “dialogues with the dead” (Fahlander and Oestigaard 2008: 4)? As frequently observed, ‘the dead do not bury themselves’ although they may continue to act through the agency of others (Erasmo 2012: 61-104). Mortuary rites are, therefore, the products of a series of considered and socially significant decisions brimming with “patterned behaviours [and] symbolic action” which need to be deciphered (Pader 1980: 143). To achieve this without straying into over-interpretation and imposing our own views onto the past, a holistic approach is required (Chapman and

Randsborg 1981; Morris 1992: 14). Evidence relating to both aspects, the necessity of dealing with the decaying corpse and the desire to signify the social being, should be addressed through the deconstruction of material components, recreation of processes and reconstruction of burial practices (Stutz 2008). Termed ‘archaeothanatology’, the methods by which such aims can be achieved (the *anthropologie de terrain* approach) have been developed and demonstrated by Duday (2009: passim) and co-workers. Consideration of the sensory impact on the survivors of the substances identified as having been employed in this transformative process should, likewise, be incorporated within this approach (Graham 2011).

The issue of bias as a corollary of differential survival must also be addressed through evaluation of taphonomic changes, some of which will have followed predictable pathways (Duday *et al.* 1990; O’Shea 1981). Ultimately, all of this evidence should be situated within its cultural context and comparisons made at different economies of scale (Chapman and Randsborg 1981). This requires consideration of the specific rite/burial ground in relation to its regional/provincial setting and, in Roman terms, its Empire-wide associations with reflection on how the discourse revealed by the materials utilised relates to socially-motivated action (Barrett 1991; Fahlander and Oestigaard 2008). Moreover, personal experience has revealed that conflicts may occur between the expectations of society, the needs of the immediate survivors and the wishes of the deceased, focussed around the treatment of the body. Thus, potential contributions at each of these levels should be explored so that individual choice is not overlooked.

2.3 Roman responses to death

The seminal work on 'Death and Burial in the Roman World' is that of Toynbee (1996). Predominantly focussed on cemetery layout and tomb architecture, it also contains a succinct survey of key aspects of Roman funerary practices based on textual and monumental evidence (Toynbee 1996: 33-64). This overview has recently been extended and complemented by an excellent sourcebook (Hope 2007) which provides access to many of the primary texts related to the subject of Roman death. In a subsequent volume, tracing the sequence of events from the deathbed to the grave, Hope (2009) then provides a stimulating synthesis of the information contained within these classical writings and in epigraphic sources. These comprehensive reviews afford clear insights into attitudes towards death in ancient Rome so there is no need to replicate their scholarship. In the following, therefore, only those aspects of significance in terms of the disposal of the dead and which serve to contextualise the treatment of the body will be addressed. As this evidence spans the 1st-4th centuries AD, care must be taken not to create a Frankenstein's monster, a fictional composite, by conflating disparate elements from four centuries of considerable change.

2.3.1 Approaching death

Death was an ever-present part of life in the Roman world although one "only recently...embraced by...historians" (Hope 2011: xi). The dead were constantly visible due to high mortality rates but also as a result of socio-cultural choices, many of which seem to have been reflected across the Empire (e.g. King 1990: 85-87; Salway 1981: 693-707; Woolf 1998: 166-169). These include the positioning of burial grounds along the roads leading to and from urban areas and in prominent or meaningful rural positions (Pearce 1999: 92-123; Toynbee 1996: 73-100), the provision of visually, audibly and olfactorily stimulating funerary displays (Gee 2008; Graham 2011; Martorelli 2000), attendance at regular festivals incorporating feasting with the dead (Dunbabin 2010: 127-132; Jensen 2008) and the staging of gladiatorial shows and prominent public executions (Edwards 2007: 46-77; Kyle 1998). Disposing of the dead and commemorating the deceased were conspicuous public events not the discrete private affairs of the western world today (Gee 2008; Hope 2009: 152-181). This was also the case with the process of dying. An "edifying spectacle" for the onlookers (Edwards 2007: 11), the final actions undertaken in this life were fundamental in defining a person's essential nature and established their lasting reputation. The manner in which death was met was deemed a true testament of character (Hope 2007: 39-45).

Incumbent on each Roman citizen was the ideal that he/she should display *virtus* as death approached by presenting an image of calm fortitude while conquering fear and pain, putting his/her affairs in order and uttering wise or witty words to friends and family (Edwards 2007: 78-112; Hope 2009: 50-55; Noy 2011). These honourable qualities were even expected to be shown, perhaps more so, if the individual had been poisoned or was, otherwise, about to be murdered as a mark of contempt for their enemies who were made to look ignoble by comparison (Hope 2007: 31-39). In some circumstances, certain forms of suicide were considered acceptable although attitudes towards voluntary euthanasia in the face of a terminal illness were more ambiguous (Hope 2009: 57-60). Thus, the need to spend time preparing for and contemplating death is often encountered in philosophical works (e.g. *Marcus Aurelius Meditations 2.1-17* nd; *Seneca Letters 63* nd) while sentiments exhorting the reader to 'live well while we can' (e.g. *Petronius Satyricon 34* nd) are frequently found in other literary genres and on epitaphs (Dunbabin 2010: 125-127; Hope 2007: 51-53). The ubiquitous reality of mortality was not something to be avoided but rather embraced in the knowledge that a 'good death' would ensure some form of continued existence.

Inevitably, many did not live up to these high moral standards (Hope 2007: 42-45) while the vast majority of the population may have viewed matters in quite a different light (Rives 2000). Due to limitations inherent in the textual evidence, our understanding of Roman approaches to death largely derives from works written by and for educated elite males residing in and around Rome during 1st century BC to 2nd century AD (Hope 2009: 11). Even epitaphs, although they provide a far broader database both geographically and temporally, predominantly relate to those who ascribed to these social mores (at least outwardly) such as members of the Roman elite, those in their households or under their patronage, individuals involved with the administrative and military systems and others among the aspiring classes (e.g. freemen, merchants, artisans) (Hope 2009: 114-115; Woolf 1998: 99-102). In death, they are generally presented in word and image in a formulaic manner that reflects this idealised world view and so the attitudes and experiences of the general populace are simply not recorded (Noy 2011). Indeed, the beliefs of the masses were frequently denigrated as mere superstition by classical authors (Rives 2000).

Likewise, insights into the changes, if any, in views on how death should be faced in the later Empire and the impact of eastern cults are somewhat elusive (Hope 2009: 111). The rise in popularity across the Roman world of religions from the east is well attested, culminating in the extensive influence of the cult of *Sol Invictus* (3rd-4th century AD) and adoption of Christianity as the official state religion around 312 AD (Esmonde Cleary 1989: 125; Potter 1987: 186-190). However, as Christian thought developed and gained sway, various groups (e.g. Donatists in Africa, Pelagians in Britain) evolved different interpretations of Jesus' teachings (Harries 1992; Raven 1993: 169-181). Asa a result, early Church leaders such as Augustine of Hippo sought to regularise Christian tenets and counteract pagan influences (Ando 2003). For example, in the '*City of God'*, he sets out the idea that although death of body comes to all as a result of original sin second death, that of soul, applied to nonbelievers only (*St Augustine 13:1-7* nd). Thus, although a greater emphasis was placed on faith and the spiritual dimension of death in terms of individual salvation, the underlying paradigm, that this rite of passage should be embraced, remained (Harries 1992). Indeed, the crucifixion of Christ and the manner in which Christian saints and martyrs faced their ends epitomised, in many ways, the concept of the 'good death': "*they show such behaviour as is adopted by philosophers; for fearlessness of death and the hereafter is something we witness in them every day*" (*Ibn Abī ʾUṣaybiʿa Galen 151* nd).

Despite the location of observance at the core of Roman culture and identity, the precise nature of the interaction between ritual action and religious thought is also hard to ascertain (Beard *et al.* 1998a: 43-54). Intrinsically an ancestor cult (Toynbee 1996: 34-35), Roman traditions were based around interpreting signs and taking the correct steps to gain divine support in order to establish a positive relationship with the gods (Ando 2003; Potter 1987: 172-173; Rives 2000). Derived from a fusion of Etruscan and Greek influences and inclusive of the deities of the conquered (Beard *et al.* 1998a: 1-72; Toynbee 1996: 11-17; Woolf 1998: 214-215), this syncretic melting pot incorporated a diverse range of views on what the afterlife, if there was one, had to offer (Beard *et al.* 1998a: 289-291; 1998b: 235-238). Nonetheless, certain constants can be identified. Key among these was "an ancient, deep-seated belief...[in the]...conscious existence for the soul after death" (Toynbee 1996: 34) and the retention of some degree of individual identity, except among the followers of Epicurus (Hope 2007: 211-247). As in Virgil (*Aeneid 6* nd), the ancestral dead were generally depicted as shadowy beings whose conduct in life affected their final destiny both in the otherworld and in the sphere of remembrance (Hope 2009: 107-111; Toynbee 1996: 34-38). These views appear in the literature from Plautus (3rd-2nd century BC) to Plutarch (1st-2nd century AD) with increased emphasis on a more concrete afterlife developing during the later Empire due to the rise of the 'mystery' cults including Christianity (Beard 1998a: 289-291, 332-335; Edwards 2007: 218-220; Hope 2009: 1-8, 111).

Of significance in terms of this study, is that fact that this "system of embedded symbols and social actions" (Ando 2003: 12) resulted in a complex web of contractual obligations between humanity and the gods through the correct enactment of contextualised ritual action (Scheid 1999). Communicated indirectly through physical manifestations (e.g. patterns in nature or evidence from sacrifices), artefacts and ecofacts were key in mediating human-divine boundaries and had a significant role to play during rites of passage (e.g. birth, marriage, death) (Ando 2003). The core of Roman eschatology "lay in ritual, not in belief: [and so] it was what people did that was important" (Rives 2000: 251). The focus as death approached fell on material rather than spiritual preparations, as recorded by classical authors and reflected in wills and on epitaphs (Hope 2009: 18, 37; Houghton 2011; Rives 2000). For an individual to achieve the entire 'good death' package, however, the setting of these actions was equally important with the ideal situation described as a death at home and/or among friends and family so that the manner of passing would be witnessed (Hope 2007: 93-95). Such an end also provided the best opportunity for the survivors to act in a manner that would serve to ensure the deceased's successful passage to the afterlife (Noy 2011).

2.3.2 Negotiating the afterlife

In the Roman period, the correct procedures for helping the deceased negotiate this significant boundary can be compiled from brief comments in a range of classical writings and a few bas-reliefs on funerary monuments (Hope 2009: 71-77). Although precise details are lacking, the ideal appears to have been for the final breath to be caught by a kiss, the eyes closed and the body placed on the ground, mirroring rites at birth (Graham 2011). The name of the deceased was called, repeatedly, amid lamentations and the body washed, anointed, dressed in garments appropriate to status and gender, wrapped in a shroud and displayed in the atrium of the house, feet towards the door, on a funeral couch surrounded by flowers and burning incense (Hope 2007: 93-99). This phase of the *funus* lasted up to seven days (Toynbee 1996: 43-45) and allowed time for the death to be announced, the body to be viewed, a death mask/portrait to be completed (although the details of this process are obscure) and preparations for the funeral to be made (Noy 2011). Throughout, the doors of the house would be marked with cypress boughs to denote a *familia funesta* whose members were prohibited from a range of social and religious activities in order to contain the pollution of death and avoid offence to the gods (Erker 2011).

The funeral itself involved an extended procession with family and friends supplemented by hired mourners and musicians accompanied by burning incense and torches so that all would be aware of its passing and could join the entourage (Erker 2011). Those of wealth and status were transported on a bier draped and canopied with purple-dyed, gold-embroidered cloth and any emblems of office were prominently displayed (Hope 2009: 74-77). When deemed appropriate, public eulogies were held, with speeches made by actors bearing *imagines*, masks representing prominent ancestors, who embodied the "central concept of post-mortem honour, both individual and collective" (Brooke 2011: 104). These obsequies were followed by a liminal feast for the living and the dead at the graveside with the pouring of libations (e.g. water, wine, milk), deposition of other offerings (e.g. grain, incense, grave goods) and casting of earth over the deceased as a mark of separation (Gee 2008; Hope 2009: 85-88). After a nine-day period of mourning and a series of rites to purify the house and mourners, a further feast was held at the tomb to mark the return of the survivors to the renegotiated social order (Erker 2011; Toynbee 1996: 50-51).

This composite account only reflects the circumstances associated with funerals that were deemed worth reporting, generally those with poetic or political resonance, and focussed on the treatment of members of the social elite due to their fame or notoriety (Hope 2009: 89-93; Houghton 2011). These were not the more mundane aspects of mortuary rites, the commonalities attendant on the remains of the majority of the populace across the Roman Empire and contrast sharply with the few references that exist concerning the treatment of the poor (Erker 2011). The latter, or those deliberately treated with disrespect, were carried on a *sandapila* (cheap bier) with little ceremony and often ended up un-memorialised in mass graves (Hope 2007: 101-102; Noy 2011): *"Let there be no long procession of my family portraits, no trumpet bewailing my fate in vain...no bier spread with ivory headrest, nor let my corpse lie on an Attalic couch. Let there be no procession of incense bearers, but only the humble rites that mark a poor man's funeral" (Propertius Elegies 2.13b: 3-8* nd). Indeed, there is little evidence of public provision for those without the means to cover funeral costs (Patterson 1992) although remaining unburied was considered the ultimate disgrace, a fate worse than death that was fraught with danger for both the dead and the living (Noy 2011). Those of lower social status, therefore, often joined burial clubs or participated in patronage relationships to ensure they would receive the essential rites and be commemorated in some manner (Hope 2007: 87-89; Osiek 2008; Patterson 1992).

The deposition of the body took place within burial grounds located outside the sphere of the living as specified in Roman law (Jones 1987) with certain exceptions (e.g. infants) (Esmonde Cleary 2000). A range of options regarding the manner of disposal seems to have been available, from simple earth-cut graves for individual cremations or inhumations to elaborate funerary monuments incorporating single or collective interments (Toynbee 1996: 101-244). Likewise, the choice between cremation, the *mos Romanus* (Roman custom), and inhumation, the *antiquissimum sepulturae genus* (ancient rite of burial), appears to have remained on offer throughout Roman history (Hope 2007: 107-111; Toynbee 1996: 14-17, 39-40). In general terms, those cremated were transported on a bier to the pyre, either an *ustrinum* (used repeatedly) or a *bustum* (above a specific grave-pit). Grave goods and offerings might be added and then the soft-tissues would be consumed by the flames in a process that would have taken much of the day (Hope 2009: 82-84). Afterwards, the skeletal remains were collected and placed in a container (anything from a cloth bag to a gold casket) for interment, often accompanied by additional, unburnt grave goods (Jones 1987). Those who were inhumed were commonly laid supine, extended in a shroud and/or wooden coffin often with a coin to pay for safe passage in the mouth or hand, articles of footwear and artefacts such as ceramic vessels, glassware and items of personal adornment (Pearce 1999: 163-164; Toynbee 1996: 48-49). A diverse range of other treatments including prone burials, decapitations and plaster burials are, however, encountered in many burial grounds (Philpott 1991: 222-226). More elaborate containers have also regularly been identified, in the form of tile-built structures, lead coffins/liners and large stone sarcophagi, sometimes located within vaults or mausolea (Ramm 1971; Salway 1981: 700-705).

Adopted at varying rates and to different degrees, these overarching mortuary practices have been observed across the provinces of the Roman Empire with inevitable variations due to local traditions (Pearce 2000). Nonetheless, the apparent provincial conformity with Roman practice in this conservative social arena is somewhat surprising and is most clearly reflected in the trend from cremation to inhumation that began in the 2nd century AD (Morris 1992: 46-68). With the exception of certain provinces with particularly strong previous traditions (e.g. Egypt) and small regional pockets (e.g. the territory of the Durotriges in Britain) where cremation was never favoured, this transformation in burial practices can be traced across the

Empire and beyond (Esmonde Cleary 1992; Parker Pearson 1993; Riggs 2010). Thus, by the end of the 3rd century AD, the normative inhumation rite described above was prevalent and remained so, in those areas where Christianity continued to flourish, until the 20th century AD. The rationale and meaning of this shift in the dominant method of body disposal has been extensively debated (Hope 2009: 81-82; Morris 1992: 40-41) but was probably related to perceptions regarding a more meaningful afterlife in conjunction with social strategies for the maintenance of elite identities (Parker Pearson 2003; Salway 1981: 698-699). It has been suggested, therefore, that correspondence across the Empire with these trends in burial practices was part of 'being/becoming Roman' and required neither a fundamental change in belief nor materialised emphasis on religious affiliation in the mortuary sphere (Harries 1992; Green 2008; Rives 2000).

The benefits in archaeological terms of this widespread change to inhumation are that a wider range of variants in the finer details of mortuary practices can be discerned, often reflecting a degree of syncretism between Roman and pre-Roman rites in different provinces. Examples range from the portrait mummies of the Fayum with their combination of Egyptian, Greek and Roman processes and iconography (Corcoran and Svoboda 2010) to barrow burials in eastern England with their range of imported luxury items (Struck 2000). Viewed in the Republican period as having specific cultural connotations (Hope 2007: 107-108), over time, certain aspects (e.g. the application of plaster body-casings) appear to become more widely incorporated within Roman practice and are found dispersed across the Empire (Counts 1996; Morris 1992: 68; Ramm 1971). The apparent south/east to north/west directionality of most of these trends has led to associations with the rise of the 'mystery' cults and has been proposed as an indicator of their influence on both religious thought and ritual practice (Sparey Green 1977; Jones 1987).

Based on these observations, certain key features can be identified. In the Roman world, the period between death and burial was marked by a series of ritual acts constrained by aspects of the deceased's social identity: age, gender, marital status and standing in the community (Hope 2009: 67-68). There is, however, little evidence to indicate that personal religious choice had a significant impact on attendant mortuary rites (Jones 1987; Harries 1992; Hope 2009: 77-79). Just as in the UK today where those from nihilists to the devoutly religious tend to be cremated with minimal memorialisation, the broad range of belief systems and views on the afterlife expressed in classical texts appear to have been concealed by adherence to social norms (Rives 2000). Thus, across the Roman Empire, most extant burials from a particular time and place conform to the dominant disposal rite with continuity in the material package used to demarcate differential status extending across the cremation-inhumation boundary (Counts 1996; MacCormack 2003; Struck 2000).

This importance of signifying the social standing of the deceased is reflected in Roman state regulations regarding the type of *funus* deemed appropriate in terms of public engagement, mode of dress of the deceased, level of conspicuous consumption and even the displays of mourning permitted (Erker 2011; Patterson 1992; Toynbee 1996: 43-61). Indeed, ‘The Law of the Twelve Tables’ (c. 450 BC) stipulates that, “*anointing by slaves is abolished...there shall be no costly sprinkling, no long garlands, no incense boxes...a myrrh-spiced drink...shall not be poured on a dead person*” (*Anon Table Ten* nd). These tenets do not seem to have held much sway by the early Empire (**3.2**) but this passage demonstrates that, although many of these status symbols were momentary and ephemeral, they were materially-manifested. Some aspects are, therefore, amenable to archaeological interrogation. For example, the type of burial container, range of more durable grave goods and, in favourable taphonomic conditions, the nature of textiles, dye-stuffs and botanical remains. Fundamental to this riot of sights, sounds and smells which surrounded the deceased from death to burial was the desire that this final rite of passage would be rendered individually memorable but would also be collectively situated through associations with ancestral traditions. The materiality of each death served to revitalise the memory of those who had gone before.

2.3.3 Maintaining memory

In the Roman world, this maintenance in memory of the *fama* (reputation) of the deceased appears to have been of paramount importance (Graham 2011; Hope 2007: 71-79). Established in life and denoted by the manner in which death was approached, there is considerable evidence to support the contention that Virgil's 'immanent shades' (*Aeneid 6.290-901* nd) were sustained by continued interaction with the living (Toynbee 1996: 61-62). Thus, preparations for death were in effect "preparations for the perpetuation of memory" (Hope 2009: 39). To this end, a range of social strategies were employed to engage the survivors in individual and collective acts of remembrance, even if they had been strangers in life: *"You traveller, who make your way along the path, Stop I ask – I beg you do not ignore my epitaph"* (*inscription, Pesaro, Italy*, cited in Hope 2009: rear cover). These concerns can be traced through instructions left in wills, involvement in burial clubs, sentiments expressed on epitaphs and the structure and location of funerary monuments and burial grounds (Gee 2008; Hope 2007: 63-70; Patterson 1992). In the house, *imagines*, death-masks or portraits of those who had achieved high office (e.g. had held magistracies) were displayed in the *atrium*, the public area of the dwelling (Noy 2011). Offerings to the deified ancestors were also made at the household shrine on a daily basis while, during the *Lemuria*, these potentially hostile spirits of the dead visited the arena the living and needed to be placated (Hope 2009: 99-100).

Most acts of commemoration were, however, undertaken at the tomb. The role of these monuments in the maintenance of memory was enshrined in Roman law (Carroll 2011) and their neglect or desecration was a matter of considerable concern (Hope 2007: 165-171). The iconography and inscriptions with which many were adorned appear to have been "intentionally chosen...to negotiate and display status and to commemorate...[the] network of personal relationships...enjoyed" by the deceased (Carroll 2011: 134-135). That some aspect of the dead dwelt within the tomb and required succour and sustenance is supported by financial and patronage arrangements stipulated in wills for the regular provision of food, libations and offerings (Erker 2011; Toynbee 1996: 37-38). The presence of libation tubes in conjunction with the design and decoration of these ‘houses of the dead’, many of which were furnished with seating, washing and dining areas to facilitate memorial feasts, likewise, emphasises the significance of continued contact (Jensen 2008).

Moreover, the Roman ritual calendar made provision for frequent visitation during official festivals such as the *Parentalia, Lemuria* and *Rosalia* (Beard *et al.* 1998a: 31, 50; Toynbee 1996: 63-64). Fundamental to these celebrations, through the re-enactment of aspects of the funeral (e.g. torches, offerings of incense, garlands of flowers, feasting), was the element of public display. A refreshment (*refrigerium*), in literal and metaphorical terms, of the relationship between the living and the dead these highly visible gatherings of friends and family served to reinforce collective memories and community ties (Gee 2008). In addition, the lighting of lamps at the graveside took place on Kalends, Ides and Nones of each month and more personal, private, tributes were made on significant anniversaries such as the deceased’s birthday (Erker 2011; Toynbee 1996: 63-64). Literary and archaeological evidence indicates that these traditional graveside rites continued even after Christianity had become widespread and were part and parcel of Roman observance across the Empire (Jensen 2008; Osiek 2008). A "dynamic performance enacted to articulate and fix proper relationships between the living and the dead" (Gee 2008: 63), feasting at the graveside was another facet of the iterative spectacle surrounding death (Hope 2009: 85-88). The relevance, in terms of the current study, of this emphasis on continued interaction with the deceased (which linked private and public spheres in the renegotiation of social structure) is that it highlights the significance placed on promoting memory through materialised ritual action in the Roman period.

2.4 Summary

Approaches to dying and the dead in the Roman world focused on establishing and maintaining the reputation and memory of the deceased. In death, even more so than in life, it seems that it was an individual’s correspondence with (or deviation from) the idealised image of the good citizen, husband/wife, parent/child that merited comment. Reinforced by the rituals undertaken during the *funus*, it was crucial for correct procedures to be carried out to denote the respect in which the deceased was held by the living and to ensure that the journey was successful. Continued interaction through memorial feasts and the provision of offerings, principally at the tomb, was fundamental in sustaining the dead and perpetuating their memory, individually and collectively. As roles in these rituals were assigned according to age, sex/gender and status, the treatment of the body mirrored, reinforced and re-negotiated fundamental aspects of Roman social structure. Continuity in these processes can be traced throughout the Empire regardless of changes in the dominant method of disposal and religious affiliation.

Thus, the text of Roman death was designed to establish the credentials of the deceased in this world in order to negotiate entrance to the next and create an enduring image of the dead through ritual action. Key in terms of this research agenda is the weight of evidence which shows that material culture was a significant factor in these negotiations. The manner in which the body was dressed and prepared, the nature of the burial container and/or tomb monument and the level of conspicuous consumption during the funerary period embodied the social standing of the deceased and acted as trigger for memorialisation. One factor in this socially embedded desire for immortality through remembrance appears to have been the materiality of aromatic plant exudates which are mentioned at every stage of the proceedings. In all of this, however, it is the treatment of the body that requires the closest attention (c.f. Hertz 1960).

Chapter 3

Treating the body: literary and archaeological evidence

"the corpse is next washed, anointed with the choicest unguents to arrest the progress of decay, crowned with fresh flowers and laid out in sumptuous raiment...bravely attired and gloriously garlanded, [he] reposes upon his lofty bier, adorned as it were for some pageant". Lucian, On Funerals (Of Mourning):*11-15* nd

3.1 Introduction

A "defining and central element of Roman identity" (Hope 2009: 94), death and its attendant rites were designed to provide a lasting image of the deceased. The final period of interaction between the survivors and the corporal remains would have been central to this socially embedded desire for immortality through remembrance (Graham 2011). Traditionally the domain of women, although increasingly placed in the hands of funerary professionals (e.g. *libitinarii* and *pollinctores* who prepared the deceased for burial) during the Imperial period, the finer details regarding the treatment of the body are difficult to ascertain (Hope 2009: 69-70; Noy 2011). References appear in a range of literary genres but, due to poetic licence or the view that these ritual acts were common knowledge, specific information is lacking (Hope 2007: 99, 108; Houghton 2011). It is also possible that this aspect of mortuary practice was perceived as an indelicate or taboo subject. Direct contact with the dead was certainly proscribed as attested by the social restrictions placed on those involved in preparing the deceased for burial and on the household during the *funus* with subsequent cleansing rituals required of the survivors (Graham 2011; Hope 2009: 94; Toynbee 1996: 50-51).

Any substances applied to the decaying corpse would have been fundamental in providing the final sensory impact of the deceased on the living and in signifying the style in which the dead had chosen (or had been chosen by others) to meet their maker (Graham 2011). Thus, in this chapter, Roman literary sources from the late Republic to the end of the Imperial period are interrogated, despite their spatial, temporal and social limitations, for insights into the use of plant exudates in the preparation of the body and indications as to those deemed appropriate for use in the mortuary sphere (**3.2**). This is supplemented by a review of the published data, obtained from reports of ‘mummified’ or ‘embalmed’ individuals (outside Egypt) and the analysis of organic residues recovered from Roman mortuary contexts, in order to establish which botanical materials have been identified and the circumstances in which they have survived (**3.3**). These findings are summarised in **3.4**.

3.2 Primary sources

3.2.1 References to scented substances

Evidence contained in the primary sources suggests that aromatic plant products played an important role in the mortuary sphere and were utilised at every stage of the *funus*. Soon after death, the body of the deceased was treated with perfumed oils or balms: *“the corpse is next washed [and] anointed with the choicest unguents*” (*Lucian, On Funerals 11* nd). Incense was burnt around the funerary couch and, probably, during the procession to the pyre or tomb as depicted on bas-reliefs from funerary monuments (Hope 2009: 74-75), although the literature is ambiguous on this point. What is clear, is that spices and other scented substances accompanied the body as it was carried through the streets with excess offerings transported on additional biers, by incense bearers (alight?) or even fashioned into effigies: “*the women contributed such a vast quantity of spices...that, apart from what was carried on 210 litters, a large image of Sulla himself...was moulded out of costly frankincense and cinnamon”* (*Plutarch Lives Sulla 38:2* nd).

If the individual was cremated, these materials were burnt along with the body: *“a costly pyre heaped with incense to give off a rich smoke of eastern perfumes*” (*Lucan, Civil War 8:729* nd). Even favoured pets might be treated in this manner. For example, Melior’s parrot was burnt: “*with spicy eastern oils...while fine and dainty feathers exhale[d] the scent of myrrh from Arab lands and Sicily’s saffron”* (*Statius Silvae 2.4:43-46* nd). It also seems that aromatic offerings could be added to the ‘ashes’ after cremation was complete and deposited within the tomb (Alcock 1980: 62; Philpott 1991: 18) so that the bones should not be consigned “*unscented to the urn”* (*Persius Satires 6:34* nd).

These practices may have continued once inhumation became the prevalent method of body disposal since the sumptuously adorned remains of the young man described by Lucian were destined to "*presently decay, or (if such is your good pleasure) be consumed with fire*" (*On Funerals 18* nd). Likewise, the funeral procession of Priscilla, with its long list of accompanying aromatics, ended with her inhumation: "*age will no longer wither you, nor will the effects of the years do you harm: such care is taken of your body, such are the riches that the venerable marble breathes*" (*Statius Silvae 5.1:228-231* nd). The latter account implies that the manner in which she had been prepared for burial would preserve her remains and may record a case of 'true' (i.e. Egyptian-style mummification; Counts 1996). It could be argued, however, that this description more closely reflects the process defined by Chioffi (1998: 24) as 'embalming' in which natural products were used to temporarily delay soft tissue degradation (with no textual or physical evidence for artificial desiccation and/or evisceration). Unfortunately, the precise nature and extent of this practice cannot be gleaned from the literature although knowledge of the various methods of body preservation utilised by other ancient cultural groupings is evident (Chioffi 1998: 22-23; Hope 2007: 107).

Characterised pejoratively as a 'foreign' practice in the late Republic and early Empire, only a small number of references to such treatments are extant. In addition to the rites accorded Priscilla, these comprise Marc Antony's (30 BC) mummification in the Egyptian fashion in Alexandria under the auspices of Cleopatra (Cassius *Dio 51.11* nd), the riches lavished on Poppaea (65 AD), the wife of Nero, who was "*stuffed with spices and embalmed in the manner of foreign potentates"* (*Tacitus Annales 16:6 nd*) in Rome, and the embalming of the Byzantine emperor Justinian I (565 AD) "with honey, balsams, unguents and incense" in Constantinople (Chioffi 1998: 23). These examples provide a commentary on the treatment of the body throughout the Roman period and support the use of resinous exudates as part of the process. Nonetheless, these exotic eastern rites appear to differ fundamentally from the descriptions of the anointing of the corpse with scented balms for display in the *atrium* (*Persius Satires 3:104-105* nd) and also from the "*fragrant forests of incense*" (*Statius Silvae 2.6:85-88* nd) piled on the bier and burnt with the body. Moreover, when the deceased was inhumed, the ultimate fate of the latter remains obscure as only two of the accounts cited (Lucian's satire *On Funerals* nd and Statius's eulogy on Priscilla, *Silvae 5.1* nd) imply that they passed into the tomb with the dead.

Details provided by Jewish sacred texts, which describe a sequence of rites comparable to those of Rome, add some support for the latter view (Green 2008). They stipulate that the body of the deceased should be purified (water), anointed (oil) and sprinkled with perfumes, funerary spices and/or dried herbs (Hachlili 2005: 376, 384). In addition, there is evidence to suggest that aromatic substances were burnt as incense during the funerary procession, sprinkled onto the bier and deposited within the tomb, with *unguentaria* (ceramic or glass vessels thought to have held scented oils or unguents) common finds in these rock-cut burial chambers (Hachlili 2005: 376-385; Green 2008). Heavily influenced by both of these traditions, Christian practices appear to have been conducted along similar lines with inhumation the preferred method of disposal (Hope 2009: 82). Thus, although there may have been sub-groups within burial populations (e.g. Christians around the tombs of martyrs, in donated/purchased plots or in areas of the catacombs), it seems that the dead were generally interred side by side with archaeologically indistinguishable rites regardless of their religious beliefs (Harries 1992; Toynbee 1996: 234-244). This is reflected in complaints voiced by the Christian Fathers about the persistence of pagan status symbols which they viewed as idolatrous (e.g. floral crowns) or ridiculed as superstitious nonsense (de Santis 2000; Martorelli 2000): *"If a rich man is buried with perfumes he may delay bodily corruption but will he not still decay?"* (*St Augustine Sermon 177: 7* nd, author's translation).

3.2.2 Indications of source

Evaluation of the primary sources suggests that a relatively limited palette of scented substances was considered appropriate for use in the mortuary sphere. Nine plant products are named with varying degrees of specificity, *amomum,* balsam, cassia, cherry, cinnamon, frankincense, myrrh, nard and saffron, alongside generic references to products associated with various ethnic groups or geographical regions (**Table 3.1**; full quotations and references are given in **Appendix 1**). In addition, the Gospels refer to the use of nard, myrrh and aloes as part of Jewish burial customs (*Bible: SEV* 2009 *Mark 14:3-8, John 19:29-40*). Of these, cherry, is mentioned only as an adulterant and appears to be used as a literary device (*Persius Satires 6:36* nd). The remainder represent some of the most desirable spices and plant exudates of the ancient world. Their sources, exploitation and uses during the early Empire are described by Pliny the Elder (*Naturalis Historia* (*NH*) nd) supported by evidence from the *Periplus Maris Erythraei* (*PME*) an account of trade routes around the Red Sea in the 1st century AD (*Anon PME* nd).

Those most frequently mentioned are cassia, the bark of *Cinnamomum* spp. (e.g. *C. iners*), and the closely related cinnamon (the inner bark of *C. verum*). In antiquity, the origins of these expensive products were surrounded in mystery (Dalby 2000: 36-39) with writers such as Theophrastus (4th century BC) stating that *"cassia and...cinnamon are found in the Arabian peninsula"* (*Theophrastus 9.4:2* nd). According to Pliny, however, these spices were purchased in Ethiopia (*NH 12.42-44* nd) while the *PME* (*Anon 8-13* nd) mentions Somali ports but also provides evidence of links with the Indian sub-continent which would have enabled 'true' cinnamon to reach the Mediterranean (Langenheim 2003: 283-290). Moreover, since certain balsamic resins (e.g. styrax and storax) contain cinnamic acid and related compounds, the well-attested vagaries in ascribing botanical origins (Groom 1997: 55-56, 65-66) mean that the bark or exudates of other species could be included under this heading. This complexity is implied by terminology such as *"rich Sabaeans' special cinnamon"* (*Statius Silvae 5.3:56* nd) and the stipulation for *"Syrian cinnamon...*[as well as]*...cinnamon bark"* (*Pliny NH 13.2:18* nd) in the royal unguent blended for the kings of Parthia.

As regards frankincense and myrrh, due to their widespread and continuing appeal, these gum-resins are the best known of those listed. Although each are only specifically mentioned twice, they would have instantly sprung to mind when reference was made to the riches of Arabia and Sabaea as they were reason the region gained the epithets '*felicis ac beatae*' (happy and blessed) (*Pliny NH 12.30:51* nd). The exudates of *Boswellia* spp. and *Commiphora* spp. respectively, extensive details of their exploitation are provided by Theophrastus (*9.4* nd), Pliny the Elder (*NH 12.30-32* nd) and a number of other classical authors (Groom 1981: 55-76). Of considerable significance in the ritual practices of many ancient cultures, large quantities were transported overland both east to Persia and north to Gaza in Judaea for use as incense and as ingredients in the holy anointing oil of the Jews (*Bible: SEV* 2009 *Ex 30:22-28; Lev 2:1;* Groom 1981: 13-20). Indeed, to add to their mystery, tall stories were recounted about the origins and collection of these highly desirable substances (Groom 1981: 58-60). Of relevance perhaps to their mortuary associations, is the myth that myrrh, alongside nard, cinnamon and cassia were used to build the nest of the Phoenix who died in its conflagration only to rise again from the ashes (as related by Ovid, *Metamorphoses 15:391-417* nd).

On a more practical note, the maritime network recorded in the PME (*Anon 8-13, 27* nd) reported that myrrh could be purchased in Somali ports and supplied considerable information regarding the 'Frankincense Kingdom' (southern Arabia). Here the best quality *Boswellia* spp. product, described by Dioscorides as that which is "*white...fat...and burns readily*" (*1.81:3-4* nd), could be obtained through the ports of Sumhuram (Dhofar, Oman) and Qana' (modern Bir Ali, Hadramawt, Yemen). Stored and sorted under draconian conditions in Alexandria for redistribution, the demand for frankincense was such that two harvestings were required each year and, although much was burnt as temple incense, far greater quantities were employed as part of funerary rites when: "*by the luxury of mankind even in the hour of death...they burn over the departed the products which they had originally understood to have been created for the gods*" (*Pliny NH 12.41:83* nd). Although myrrh was also burnt as incense, it seems that it was more commonly used as an unguent or perfume, either alone (its initial exudate known as *stacte*) or as a stabilising agent in complex mixtures, due to its ability to maintain scents over extended periods of time through the retention of more volatile components (*Pliny NH 12.33-35, 13.2:7,17* nd).

Table 3.1. Primary sources that specify botanical source or geographical place of origin of resinous substances used in the mortuary sphere (see **Appendix 1** for full references).

Source	Date	Original term/phrase	Translation (by author)
Petronius *Satyricon*	1st c. BC	ampullam nardi	Jar of nard
Propertius *Elegies*		Nardo	Nard
		Syrio munere	Gifts of Syria
Tibullus *Elegies*		Assyrios...odores	Assyrian scents
Pseudo-Tibullus *Elegies*		dives Panchaia merces Eoique Arabes, dives et Assyria	Riches and gifts of Panchaia, the East, Arabia, and Assyria
New Testament *Gospels*	1st c. AD	ναρδου	Nard
		σζμυρνης και αλοης	Myrrh and aloes
Plutarch *Lives, Sulla*		ἀρωμάτων λιβανωτοῦ κινναμώμου	Perfumes Frankincense Cinnamon
Persius *Satires*		Cinnama Casiae Ceraso	Cinnamon Cassia Cherry
		crassisque lutatus amomis	Thick and [?] amomum
Statius *Silvae*	Late 1st c. AD	Arabum Cilicumque fluit floresque Sabaei Indorumque arsura seges... tura Palaestinis, simul Hebraeique liquores Coryciaeque comae Cinyreaque germina	Produce of Arabia and Cilicia Sabaean flowers Indian crops for burning Palestinian incense Hebrew liquids (perfumes) Corycian threads (saffron) Seeds of Cinyreia (myrrh)
		Cilicum flores graminis Indi Arabes Phariique Palaestinique liquores	Cilician flowers Indian spices Arabian, Egyptian, Palestinian perfumes
		Assyrio amomo Arabum respirant gramine Sicaniisque crocis	Assyrian *amomo* Aromatic crops of Arabia Sicanian saffron
		odoriferous...Sabaeos Cilicum messes Pharian cinnama Assyrio manantes gramine sucos	Scented produce…of Sabaea Cilician harvests Egyptian cinnamon Juices that flow from Assyria
		Eoa germina messes Cilicumque Arabumque	Produce of the East Harvests of Cilicia and Arabia
		Sicanii...croci, Sabaei cinnama odoratas Arabs...aristas	Sicanian saffron Sabaean cinnamon Scented Arabian grains
Martial *Epigrams*		Murram Casias	Myrrh Cassia
		Casias Murram Incense Cinnama	Cassia Myrrh, Incense (?frankincense) Cinnamon
		Amomum	Amomum
Juvenal *Satires*	1st-2nd c. AD	Amomum	Amomum
Ausonius *Epitaphs*	4th c. AD	unguine nardi balsama	Nard Balsam

From closer to Rome, the spice saffron (the stigmas of *Crocus sativus*) is explicitly mentioned on three occasions but only by Statius (late 1st century AD) as part of a sequence of substances that form a poetic trope redolent with funerary symbolism (*Silvae 2.1:214-227* nd). Associated with Corycia in Cilicia (southern Turkey) and Sicania (Sicily), when phrases such as 'Cilician harvests' appear elsewhere they are, therefore, generally interpreted as referring to saffron although balsamic exudates such as styrax and storax (possibly from *Styrax officinalis* and *Liquidambar orientalis*, respectively) were also obtained from this region (e.g. Langenheim 2003: 347-8, 354-5; *Pliny NH 12.55* nd). Part of a package of "*lavish [aromatic] gifts*" (*Statius Silvae 2.1:214* nd) garnered from a range of exotic geographical locations, the use of such generic terminology was clearly expected to resonate with the social elite at whom this poetry was directed. Indeed, Pliny refers explicitly to the concept of 'branding' by place of origin in his discussion on perfumes and to changes in fashionable preferences for certain products (*NH 13.2* nd). Thus, through consideration of the evidence provided in other literary genres some indications as to the substances alluded to may be obtained. For example, as noted above, the aromatic liquids, incense, produce, crops or harvests (*liquores, odores, germina, graminae*) of Arabia probably refer to the gum-resins frankincense (*Boswellia* spp.) and myrrh (*Commiphora* spp.) (*Pliny NH 12.30-35* nd) but could include other aromatics that were traded via their ports (*Anon PME 27-39* nd).

In the ancient world, the Levant (Roman province of *Judaea/Syria Palaestina*) was another region renowned for its scented substances with the most desirable being balsam which is widely attested in the literature from Theophrastus (3rd century BC) to Tacitus (2nd century AD) (Safrai 1994: 83-87). Generally thought to have been extracted from members of the family Burseraceae such as *Commiphora opobalsamum, C. gileadensis* and/or related species, even in antiquity there was considerable uncertainty as to the precise botanical source of this exudate which seems to have been particularly difficult to extract on a commercial scale (Howes 1949: 153; Langenheim 2003: 356-357). This obfuscation may have been deliberate as control of the balsam trade created considerable hostility between the Jews and their Roman overlords (*Pliny NH 12.54* nd).

Referred to under a variety of names (Balsam of Gilead, Judaea, Mecca or Syria), even its geographic origins were debated which suggests that more than one botanical source was almost certainly involved (Groom 1981: 126-131; *Theophrastus 9.5:6* nd). References to Palestinian incense and Hebrew perfumes may, therefore, encompass a range of plant exudates harvested in the region around Judaea. These include styrax (*Styrax officinalis*, although this species seems no longer to be productive), storax (*Liquidambar orientalis*) (Langenheim 2003: 347-355) and terebinth, probably *Pistacia terebinthus* or the closely related *P. atlantica* (Hairfield and Hairfield 1990; Mills and White 1989). In fact, the latter may have been the source of Balsam of Syria: *"in...Damascus...there is a certain hill which is covered with terebinths"* (*Theophrastus 9.2:2* nd).

Returning to the substances specifically named by Roman authors for use in mortuary rites, the earliest references, together with Ausonius' 4th century AD evocation of past practices (*Epitaphs 6.31* nd), are to nard. Described by Pliny as holding *"a foremost place among perfumes"* (*NH 12.26:42* nd), the leaves of the plant known as nard or spikenard (probably *Nardostachys jatamansi*) from which the extract was obtained were traded from the Gangetic region of India (*Anon PME 48-49, 56* nd) with other varieties of valerian from Syria, Gaul and Crete sometimes exploited in this manner (*Pliny NH 12.26:44-46* nd). This term was, however, also used to denote a *"leaf-unguent...made of omphacium* (extract of immature olives or grapes)...*behen-nut oil* (essential oil of *Moringa oleifera*)*, rush* (probably *Acorus calamus*)*, costus* (root of Indian thistle, *Saussurea costus*)*, nard, amomum* (unidentified)*, myrrh and balsam*" (*Pliny NH 13.2:14-15* nd; for details on components see, Groom 1997: 315; *Pliny NH 12.25-54* nd; *Theophrastus 9.4-7* nd).

Which form of nard, or whether either might be used, in the mortuary sphere is unclear although the literature indicates that it could be applied as part of the anointing ritual that occurred soon after death (e.g. *Bible: SEV* 2009 *Mark 14:3-8*; *Petronius Satyricon 77-78* nd) and that it could also be sprinkled on the pyre or over the ashes within the urn (*Ausonius Epitaphs 6.31* nd; *Propertius Elegies 2.13b:13-16* nd). Similarly, aloes, the leaf extract of members of the genus *Aloe* from Arabia and eastern Africa (Groom 1997: 5-6), and the unidentifiable *amomum* obtained from India or Assyria (Dalby 2000: 102-103; *Pliny NH 12.28* nd), seem largely to have been linked to the preparation of the body with the powerful scent of the latter strongly associated with the imagery of death: *"reeking...with [amomo] enough to out-scent two funerals"* (*Juvenal Satires 4:108* nd).

Thus, the primary sources indicate that a limited range of plant products were employed as part of mortuary rites throughout the Roman period. Nonetheless, these highly subjective literary allusions predominately relate to practices in Rome during the 1st century BC-1st century AD and so the situation in the later Empire remains unclear. In particular, information about the final destination of the abundance of aromatics piled around the body and carried in the cortège once the deceased was inhumed is lacking. Chemical evidence obtained through the analysis of archaeological samples is required, therefore, to consider the true nature of any extant substances employed, the form in which these resinous exudates may have been deposited (libated in liquids, burnt as incense, incorporated in unguents or scattered as solid offerings) and the extent to which these rites were adopted in the northern provinces, far beyond the geographical range of the botanical sources mentioned.

3.3 Archaeological evidence

3.3.1 Research potential

Extant descriptions relating to the treatment of the body (**3.2**) suggest that aromatic plant products, in the form of perfumes (liquids infused with scents), unguents (perfumed ointments/creams) and as part of burnt and unburnt offerings (spices, resins, gum-resins), alongside textiles (garments, shrouds, bier-cloths) played a key role in Roman mortuary practices. Representing a considerable financial investment (Hope 2009: 67-68), as demonstrated in **Chapter 2**, the significance of these materialised agents was intimately related to aspects of an individual's identity with particular emphasis on their social status as scathingly expressed by Juvenal (1st century AD): *"there are many parts of Italy...in which no man puts on a toga until he is dead"* (*Satires 3:171-172* nd). In relation to the use of textiles as part of the adornment of the dead, recent research has enhanced our knowledge of the fabrics, garments and decorative elements employed in the Roman period (e.g. Devièse *et al.* 2011; Gleba 2008; Wild 2013). This work has been facilitated by improved approaches to excavation as well as developments in imaging technologies and instrumental analysis (e.g. Mitschke and gen. Schieck 2012; Reifarth 2013: 7-10; Wild 2012).

Despite these advances, the role of plant exudates has received far less attention. This is, in part, due to the impact of degradation processes on these amorphous organic materials but also, all too frequently, on issues of archaeological recovery and retention (**6.2**). Thus, researchers have tended to focus on more substantial facets of the burial record such as tomb architecture, burial containers, grave orientation and grave goods (e.g. Philpott 1991: passim; Toynbee 1996: 73-281) or on information derived directly from the skeletal remains to create osteobiographies of individuals and address broader questions based on cemetery populations (e.g. Eckardt *et al.* 2014; Montgomery *et al.* 2010; Redfern *et al.* 2010). As a consequence, the collection of samples that could demonstrate the presence and purpose of substances applied to the body has rarely been undertaken except with regards to artificial mummification in Egypt. Observations made by antiquarians and the discovery of visible residues from Roman period mortuary contexts in Europe indicate, however, that the potential for the recovery of such evidence exists.

3.3.2 The evidence from Rome

Reports regarding Roman period inhumation burials (outside of Egypt) containing the remains of unusually well preserved individuals span the 15th to 20th centuries AD. Many of these accounts of potential 'mummification' and ‘embalming’ in continental Europe have been catalogued by Chioffi (1998: passim). She interprets the varying forms of body treatment detailed as indicative of the influence of foreign belief systems, predominantly those of Egypt, on Roman burial practices which were later adopted by Christians (Chioffi 1998: 33-41). Of the forty probable examples given, thirty derive from the environs of Rome itself and include the references in the primary sources to the funerals of Poppaea and Priscilla, mentioned above (**3.2**). The majority of the remainder were discovered prior to the 1950s with only two more recent finds (Chioffi 1998: 47-50, 53-55, catalogue nr. 10 and nr. 13, the latter having subsequently been lost). Just ten of these reports specifically mention the application of aromatic substances to the body with one additional reference to a silver box containing various ointments interred with the wife of the Emperor Honorius (nr. 3, Chioffi 1998: 36-44; **Table 3.2**).

Within this small sample, both males and females of varying ages are represented with a greater number of subadult/young adult females. All of these ‘embalmed’ individuals had been interred in marble or porphyry containers, within mausolea or vaulted tombs. Provided with jewellery and other high quality grave goods, including hair nets of twisted gold threads (in #2 burials), most were dressed in elaborate garments and wrapped in shrouds or textile bandages. Plaster was also noted in five, perhaps six (‘hardening substances’, nr. 2), of these inhumations alongside portions of hair (in #4) (**Table 3.2**). The partial gilding of the face and hands of nr. 11 and full gilding of its companion (nr. 12, which is not reported as having been embalmed) may represent rites introduced from Egypt (Chioffi 1998: 52; Zimmer 1993). Many of these descriptions suggest that the “fragrant unguents and perfumes” (Ficoroni 1732 cited in Chioffi 1998: 46) had been added over or incorporated within the garments and below the plaster, where present. This treatment seems to reflect a practice at variance with full mummification (i.e. desiccation using natron, evisceration or purging/cleansing of the body cavity, the application of substantial quantities of embalming substances to the inner as well as the outer surface of the corpse and the elaborate bandaging of the remains).

Table 3.2. Reports of residues indicative of embalming from sites around Rome, compiled from data collected by Chioffi (1998).

Chioffi catalogue number (nr.)	*Find date*/Location	Date (c. AD)	Sex/Age	Container	Body treatment
2	*1507.* Rome, Vatican. Beneath presbytery of Constantine/St Peter's.	?2^{nd}-3^{rd}	Sub-adult	Sarcophagus, marble with sealed lid and inscription.	Embalmed body sprinkled with a liquid mixture. Dressed in a robe woven with gold. Thick layer of 'hardening' substances around the limbs?
3	*1544.* Rome, Vatican. In circular mausoleum of Emperor Honorius/chapel of St Petronilla. *1544*	Late 4^{th}-early 5^{th}	Mary, daughter of Stilicho and wife of Emperor Honorius – young adult	Sarcophagus, red porphyry with lid.	Wrapped in robes of gold tissue. *No mention of embalming.* Grave goods: silver make-up box containing *various ointments* and amulets, casket of silver-gilt with many jewels, gold and precious stones, amulet inscribed with names of the archangels, needles with Mary and Honorius, golden 'bulla' with names of family members, ?a doll.
9	*1731.* Rome, Via Cassia. Vaulted tomb with plastered inner walls, near roadside.	?1^{st}	Adult female - 'Attia' ?2 children	Sarcophagus, marble lid.	"fragrant unguents and perfumes had scented the body". Grave goods: jewellery, ?amulet, gold hair net. No record of condition of body. Red-coloured hair.
10 **EXTANT ANALYSED**	*1964.* Rome, Grottarossa, Via Cassia. ***[Ascenzi et al. 1996, see below]***	2^{nd}	c. 8 year old girl *Images, Chioffi 1998: Plates 29-31*	Sarcophagus?, marble, decorated with lid.	'True' mummification [see details of subsequent analysis given below, Ascenzi *et al.* 1996] Grave goods: jewellery, make-up items, ivory 'doll'
11-12	*16^{th} c. AD.* Rome, Via Cassia.	Late 1^{st}-2^{nd}	11 - adult female 12 - sub-adult ?female	11 – sarcophagus, marble with lid + gilded interior.	Partially gilded (face and hands) and embalmed body. 'Egyptian style'. Painted portrait mask? (Toynbee 1996: 42). NB: nr. 12 was also described as a gilded mummy but with *no mention of embalming.*
13	*1967.* Rome, Via Labicana-Casilina. In structure under circular mound.	'Augustan age'	Adult male - tall	Sarcophagus, marble with lid containing false window.	Wrapped in leather and encased in preservatives. Resinous substances and plaster reported. Body poorly preserved.
15-29 **ONE ANALYSED**	*1914-1939.* Rome, via Appia Antica, San Sebastian. Excavations around the 'basilica of the apostles', 10 marble sarcophagi + various other forms (e.g. tile-built, marble slab-lined, double burials) ***[Mitschke and gen. Schieck 2012, see below]***	2^{nd} -5^{th}	16 - robust adult male 17 - young female 21 - ? 25 - female	Sarcophagus, marble, decorated Sarcophagus, marble decorated Slab-lined, marble, some from a previous burial Sarcophagus, divided - paired with another plaster burial	16 - plaster and aromatic resinous substances, areas still semi-fluid and others solidified to create a casing. 17 - thin layer of plaster, sprinkled with hydrocarbon-based substances and wrapped in textiles with gold threads. 21 - thin layer of plaster, sprinkled with balsamic substances and wrapped in bandages/shroud of linen and wool. Traces of gold threads on head. 25 – 'embalmed' with thin layer of a plaster over abdomen, wrapped in linen shroud and crossed bandages, strewn with leaves of herbs. Hair present.
30 **ANALYSED IN 1400s**	*1485.* Rome, via Appia Antica. Above ground monument, reburied on orders of the Pope.	2^{nd}-3^{rd}	12-15 years - female *Artists' impressions, Chioffi 1998: Plates 21-23*	Sarcophagus, marble with sealed lid.	Corpse coated with thick casing of transparent substances. Body perfectly/artificially preserved but quickly deteriorated. Analysis indicated that mainly comprised scented gum-resins (e.g. myrrh, frankincense, 'aloe turpentine') mixed with cedar oil and olive oil. No reference to clothing. ?Hair present.

Exceptional preservation of the body is described, however, with regards to nr. 10 and nr. 30. Indeed, an attempt was made to identify components of the mixture used in the treatment of the latter which was discovered in 1485 and reburied on the orders of the Pope. The thick casing of transparent substances reported was found to comprise some form of gum-resin (e.g. myrrh or frankincense) mixed with cedar oil and olive oil (Chioffi 1998: 66-67). More recently, the body of a girl c. 8 years old discovered at Grottarossa, Rome in 1964 (nr. 10) has been analysed using modern methods (Ascenzi *et al.* 1996; **Figure 3.1**). Probably interred in a marble sarcophagus below a funerary monument, she had been wrapped in textiles and equipped with gold and sapphire jewellery, an ivory 'doll' and items of amber (Toynbee 1996: 42). Artefact typology indicated that this burial was dated to the 2nd century AD while palynological investigations revealed plant remains which included conifer and Cistaceae pollen but also grains, possibly, from African source (perhaps a *Commiphora* spp.) within the wrappings (Ciuffarella *et al.*1998). Analysis of the substances applied to the skin, silk cloth and linen bandages demonstrated the presence of sesquiterpenes and abietic acid derivatives indicative of a conifer resin (Ascenzi *et al.* 1996).

Figure 3.1. Images of child from Grottarossa, Rome, Italy: a. appearance on discovery, drawing made from photograph; b. appearance in 1996 (Figure from Ascenzi *et al.* 1996: Figure 9, 213).

Another more recent find (1999-2000) comes from the Via Latina, Grottaferrata, outside Rome. In an area renowned for opulent Roman villas, an intact subterranean burial chamber containing two marble sarcophagi was discovered (Ghini *et al.* 2005). Decorated with bas-reliefs and inscriptions, these containers provided the names of their occupants, a mature adult female (*Aebutia Quarta*) and *Carvilius Gemellus*, her eighteen year-old son. Analysis revealed that *Aebutia* had been dressed in a silk garment, wrapped in a shroud and covered by a wool cloak. A net of gold thread had adorned her hair which was enhanced with a false braid gathered in a knot at the nape of the neck. Placed on a funerary bed, she was wearing a gold signet ring with a male head carved in rock crystal. *Carvilius* was also wrapped in a shroud, appeared to have been laid on a mattress (fibres of cotton, linen and wool were recovered) and had been garlanded with flowers. Moreover, it was suggested that he may have been embalmed as traces of myrrh and colophony (pine resin) were reported although precise information about the analytical method(s) employed were not provided (Ghini *et al.* 2005).

In addition, as part of a research project focused on the recovery and analysis of textile evidence from Rome (Mitschke and gen. Schieck 2012), samples were collected from the catacombs (*Sant' Agnese, Santi Pietro e Marcellino* and *San Sebastiano*) and a marble cinerary urn (1st-2nd century AD) recovered near the Via Ostiense. Dated to the 3rd-4th centuries AD, the materials from the catacombs were associated with bodies inhumed in shrouds and placed in *loculi* and with an individual interred in a sarcophagus in a vault beneath an early Christian church. This study revealed not only a fascinating range of textiles but also organic residues with evidence of an "oily or fatty" substance (Mitschke and gen. Schieck 2012: 121) impregnating the fringed textile woven from flax with purple-dyed wool detailing, probably used as a shroud, that was found in the cremation urn. Traces of plaster with dark layers from *loculi* burials were, likewise, interpreted as possible evidence of embalming residues although no analytical data was obtained.

The marble sarcophagus burial from *San Sebastian*, however, revealed skeletal remains on a "bed of powdery...material...with the bundle...covered with a hard, smooth substance of reddish or black colour" (Mitschke and gen. Schieck 2012: 126-127). GC-MS analysis of a single fragment from the head region showed traces of carboxylic acids together with cinnamic and *p*-hydroxycinnamic acid potentially indicative of the use of an unguent which included a plant oil (although the source is not discussed) or a balsamic resin (although no terpenic compounds were reported). In addition, textile fragments and plaster were recovered from a series of multiple burials found within the catacomb of *Santi Marcellino e Pietro* (Mitschke and gen. Schieck 2012). Subsequent analysis showed that gypsum that had been used to encase these individually enshrouded inhumations which were stacked in rows (Blanchard *et al.* 2007; Deviése *et al.* 2010; Kachi *et al.* 2014). Human hair, textile fragments, gold threads and a pair of gold earrings were recovered, with staining evident on some of the textiles, alongside red crystalline residues (Blanchard *et al.* 2007). Analysis of the latter indicated that the source was a conifer resin, probably sandarac together with what appeared to be amber, a fossilised diterpenoid resin (Deviése *et al.* 2010).

3.3.3 Finds in the provinces: Italy and Greece

Examples of the use of resinous substances in the treatment of the body have also come to light in the Roman provinces (**Table 3.3; Table 3.4**). Chioffi's (1998: 68-69) volume describes a particularly interesting discovery from Ferento, in Etruria which was dated to the 7th century BC. This comprised two sarcophagi containing remains interpreted as those of a male interred with iron weapons and an 'embalmed' female accompanied by copper-gilded bronze utensils and containers in a variety of materials (copper, terracotta and glass). No further information as regards the nature of the body treatment was supplied but this provides a potential precursor from an area of Italy which had a considerable impact on subsequent Roman mortuary and religious practices (Toynbee 1996: 11-24). Although brief references of this nature cannot be verified and must be approached with caution, more recent discoveries outside Rome again provide some support for antiquarian observations regarding the presence of resinous substances in a number of mortuary contexts.

Table 3.3. Reports of residues indicative of embalming from sites outside Rome, compiled from data collected by Chioffi (1998) and Reifarth (2013).

Chioffi No.	*Find date*/Location	Date	Sex/Age	Container	Body treatment
31	*16^{th} c. AD* Ferento, Etruria	7^{th} c. BC	Adult female	Sarcophagus	Referred to as 'embalmed' Grave goods: bronze utensils, copper, terracotta and glass vessels. Alongside sarcophagus containing male with iron weapons.
33	*1756* Lugdunum (Puy-de-Dôme), Gaul (France)	?3^{rd} c. AD	Male. 10-12 years old	Sarcophagus - granite with lid and inner lead coffin	Perfectly preserved, evidence of soft tissues and organs. Preservative 'balsamic' substances with strong aroma sprinkled both over the body and the shroud and linen bandages in which the remains were wrapped.
34-36	*1912-1962* Carnuntum (Petronell, Austria) and Aquincum (Budapest, Hungary), Pannonia	3^{rd}-4^{th} c. AD	1 undetermined 3 females	Sarcophagus Slab-lined graves	Described as artificially desiccated with saline substances these now appear to be plaster burials. Some mention of fabrics soaked in resin. One with painted wooden portrait mask and plant remains (roses, cherries, apricots). All provided with high quality textiles (wool, cotton, linen, silk) and footwear (cork-soled sandals). Grave goods: jewellery, wooden boxes, hairpins, a spindle, glass vessels and coins.
38	*1970* Callatis (Mangalia, Romania), Moesia Above ground monument under a circular mound in vast Greco-Roman necropolis.	2^{nd}-3^{rd} c. AD	Female c. 50 years old. Evidence of osteoarthritis. Hair and some soft tissue remaining. Pyxis holding x2 teeth of the deceased.	Sarcophagus – marble with gabled lid sealed with mortar and inner wooden coffin.	Described as preserved by resin. Body placed on a layer of foliage (twigs, charred debris and pieces of resin) on a canvas mattress. Leaves and flowers placed in baskets or scattered over the body. Dressed in a decorated tunic and wrapped in a shroud with a veil over face and chest. Six pairs of footwear (x1 worn). Vast array of grave goods: including crown of gold oak leaves, gold jewellery, glass vials and vessels, writing implements, mirrors and items, possibly of ritual significance.
Reifarth No.	***Find date*/Location**	**Date**	**Sex/Age**	**Container**	**Body treatment**
A1-A7	St Maximin's, Trier	Late Roman		Sarcophagi (x6) – sandstone; 1 brick	1 with wood shavings, 1 ?with plaster, 3 with textiles – dark residues interpreted as leather recorded.
B1	Northern cemetery, Mainzer Straße, Worms	?	?	?	Three large pieces of fragrant resin totalling 135 grams found with calcined bones – presumably a cremation.
H1	Grave 20, Abbey of Saint Victor, Marseilles	Late 4^{th}-5^{th} c. AD	Female c. 20 years old. Significant soft tissue preservation.	Sarcophagus - decorated.	Bandaged with linen strips. Dressed in a silk Coptic-style tunic with gold foil and dyed yarn, a fringed mantle of silk twill and a short veil. A wreath of leaves had been placed around her head alongside a gold ring and cross. A mixture of frankincense (50%) and herbs had been applied to the body.
H3	*1908* Fin Renard necropolis, Bourges	3^{rd} c. AD	Infant 2-3 years old	Sarcophagus - limestone with inner lead coffin.	Dressed in plain weave wool tunic with sleeves and green *clavi* (stripes). Described as 'mummified' but recent re-investigation indicates this was natural as a result of burial conditions (Thillaud 2004; Thillaud *et al.* 2006).

Table 3.4. Results of the chemical analysis of samples from Roman period inhumations from Rome and its provinces, compiled from the literature.

Find	Publication ref.	Date	Brief details	Results of chemical analysis
Via Appia Antica Rome	Cited in Chioffi 1998: 66-67 [Analysed in the 1400s]	2^{nd}-3^{rd} c. AD	12-15 years old female in sarcophagus with marble lid.	Gum-resins (e.g. frankincense or myrrh). Cedar oil and olive oil.
Via Latina Grottaferrata, Rome	Ghini *et al.* 2005	1^{st}-2^{nd} c. AD	18 year old, Carvilius Gemellus, in a marble sarcophagus.	Traces of myrrh and a conifer resin reported (no information about method of analysis).
San Sebastian Via Appia Antica Rome	Mitschke and gen. Schieck 2012	2^{nd}-3^{rd} c. AD	Adult ?male in marble sarcophagus (possibly Chioffi Nr. 16 but no mention of plaster).	Fatty acids indicative of plant oil/animal fat. Cinnamic acid + *p*-hydroxycinnamic acid, a plant oil or a balsamic resin suggested (although no terpenic compounds are mentioned).
Via Cassia Grottarossa, Rome	Ascenzi *et al. 1*996	2^{nd} c. AD	c. 8 year old girl possibly interred in decorated marble sarcophagus.	Sesquiterpenes and abietic acid derivatives indicative of the presence of a conifer resin. Egyptian-style mummification.
Catacombs of *Santi Marcellino e Pietro* Via Casilina, Rome	Blanchard *et al.* 2007 Devièse 2008 Devièse *et al.* 2010	2^{nd}-3^{rd} c. AD	Multiple burials within pits, individually wrapped in shrouds with gypsum.	Diterpenoids indicative of the presence of the conifer resin, sandarac. Amber, based on lower molecular mass components, was also suggested although succinic acid has other sources.
Necropolis, *Università Cattolica,* Milan	Bruni and Guglielmi 2005 Bruni and Guglielmi 2014	3^{rd} c. AD	'Lady of the Sarcophagus', young adult female in gneiss sarcophagus.	Triterpenoids characteristic of *Pistacia* spp. resin.
Basilica of *St Ambrogio*, Milan	Bruni and Guglielmi 2014	4^{th} c. AD	Remains of *SS. Ambrogio, Gervasio e Protasio* in porphyry sarcophagus.	Triterpenoids characteristic of *Pistacia* spp. resin.
Eastern cemetery, Thessaloniki, Greece	Papageorgopoulou *et al.* 2009	3^{rd}-4^{th} c. AD	Mature adult female in marble sarcophagus with inner lead coffin.	Range of fatty acids + sesqui-, di- and triterpenoids indicative of scented unguent or oil.
Naintré, Gaul (Poitou-Charentes, France)	Devièse 2008 Devièse *et al.* 2011	3^{rd} c. AD	Two tomb vaults containing sarcophagi with lead liners – adult female and 12 year old child.	*Boswellia* spp. (frankincense) and *Pistacia* spp. (mastic) on and around both bodies. Black substance provisionally interpreted as *Boswellia* spp. bark located above the remains.
Anché, Gaul (Indre-et-Loire, France)	Devièse 2008	2^{nd}-4^{th} c. AD	Two sarcophagi - remains of an adult female and 12 year old child.	Pinaceae (?pine) and frankincense with the adult. *Pistacia* spp. resin with the child.
St Maximin's, St Matthias', grave of St Paulinus, Trier, Rhineland (Germany)	Reifarth 2013: 96-99	4^{th} c. AD	Individuals of all ages and both sexes (some multiple burials) in stone sarcophagi (x23 analysed) in crypts.	Pinaceae, Cupressaceae, *Pistacia* spp., balsamic resin (GC-MS); gums and gum-resins (FTIR)
Iovia, Pannonia (Hungary)	Reifarth 2013: 501-505	2^{nd}-4^{th} c. AD	Sarcophagi from the Roman period south-eastern burial ground.	'Thermoanalysis' indicated pine and frankincense. GC-MS of a double burial confirmed Pinaceae and *Pistacia* spp. resins
Palmyra, Syria	Reifarth 2013: 506-507	1^{st}-3^{rd} c. AD	Sarcophagi within tower tombs, one from tomb of Atenatan sampled.	Mixture of gum and a ?balsam – cinnamic acid + related compounds

For example, the immaculate excavation and in-depth analysis of an intact inhumation burial recovered from the necropolis of the Università Cattolica, Milan, in 1991, demonstrates the level of information that can be accessed. This young adult female, now known as the ‘Lady of the Sarcophagus’, had been placed on a stretcher and interred within a substantial gneiss container during the 3rd or 4th centuries AD. Dressed in fine fabrics and wrapped in a shroud, she was accompanied by gold foils and wires associated with beads and amber leaves (possible representative of a hair net or wreath), an ivory spindle and a fan (Rossignani *et al.* 2005). In addition, around her cranium and between the femora, remains of garlands of flowers were observed alongside “masses of a spongy material having a deep yellow colour” (Bruni and Guglielmi 2014: 615). Assumed to be frankincense, since a strong scent was released when a small portion was burnt, 6 g was taken for analysis. These amorphous deposits were identified as *Pistacia* spp. resin using a range of complementary techniques (Bruni and Guglielmi 2005; 2014). Likewise, investigation of a 5 g sample described as frankincense in 1864 and retained in the archives of the basilica of St Ambrogio, Milan proved to be *Pistacia* spp. (Bruni and Guglielmi 2014). Originally obtained from the porphyry sarcophagus in which the remains of *St Ambrogio* and two martyrs, *SS. Gervasio* and *Protasio* had been interred, this “whitish substance” (Bruni and Guglielmi 2014: 615) was found in association with cloth dyed with ‘Tyrian’ (murex) purple and decorated with gold threads.

In addition, in 1962, the partially mummified remains of a mature adult female were recovered from the eastern cemetery, Thessaloniki, Greece (Papageorgopoulou *et al.* 2009). Dated to the late 3rd-early 4th centuries AD, this individual had been placed on a wooden pallet inside a marble sarcophagus with inner lead coffin. The body had been wrapped in cotton or linen bandages and covered or dressed in purple-dyed silk decorated with gold thread. Due to exceptional taphonomic conditions, not only the head hair, twisted into a long plait, but also the eyebrows and some soft tissue had been preserved. Examination of the skeletal remains suggested that this ‘lady’ was of high status as, despite her age, only minor degenerative changes were present with no signs of poor health or an inadequate diet.

Dark residues were observed adhering to the skeletal remains (**Figure 3.2**). Chemical analysis of loose particles and materials adhering to the hair provided evidence for a range of compounds including carboxylic acids, cinnamates and sesqui-, di- and triterpenes which were interpreted as the remains of an embalming unguent, although precise molecular identification was not attempted so the botanical source(s) could not be established (Papageorgopoulou *et al.* 2009).

Figure 3.2. Vertebrae with dark organic residue, possibly soft tissue, sarcophagus burial, Thessaloniki, Greece (Image from Papageorgopoulou 2008: news release).

3.3.4 The northern provinces: Gaul and the Rhineland

In this region, the presence of resinous plant exudates in Roman period mortuary contexts appears to have been purely speculative prior to the most recent multi-analytical investigations undertaken in this field (**Table 3.3; Table 3.4**). Indeed, as demonstrated by re-evaluation of the 'mummified' infant from the *Fin Renard* necropolis (curated in *Le Musée du Berry,* Bourges) by Thillaud (2004; Thillaud *et al.* 2006), non-specific terminology and natural taphonomic processes seem to have resulted in the misinterpretation of finds. Thus, although elaborate body treatments were reported, the use of exotic aromatics had not been confirmed.

The discovery in 1997 of a vaulted double tomb at Naintré, near Poitiers, west-central France began to re-dress this issue through detailed excavation and examination of its contents (Devièse 2008: 65-68). Dated to the 3rd century AD, each crypt contained an intact interment in a lead coffin sealed within a limestone sarcophagus. The earlier of the two held the remains of an adolescent around 12 years old and the other, those of an adult female.

Textiles were present in both burials. The child, whose upper torso was covered in gypsum, had been supplied with silk damask enhanced by murex purple-dyed elements and decorative gold tapestry bands (Bédat *et al.* 2005). A range of high quality grave goods including glass vessels, an ivory writing tablet and comb, a spindle and many items in bronze also accompanied this burial and a garland of flowers had been placed on the lid of the lead coffin. In the case of the adult female, fewer grave goods were present although some hair and degraded textiles (including a damask, probably silk, and gold threads) were recovered. Lime plaster had been spread over the body alongside an abundance of powdered murex purple dye and, remarkably, traces of clay pigments, which had been applied to the face, were identified (Devièse *et al.* 2011). Analysis of amorphous organic materials from within both burials provided chemical evidence for the presence of frankincense (*Boswellia* spp.) and mastic (*Pistacia* spp.). In addition, a black substance located above the human remains was tentatively identified as *Boswellia* spp. bark.

Similarly, two stone sarcophagi with inner lead coffins were found (in 2000) as part of a group of three inhumation burials within a large quadrangular monument near Anché in the Loire Valley region of central France (Devièse 2008: 63-65). Dated between 2nd-4th centuries AD, these again comprised the inhumations of an adult and child (c. 12 years old). Preserved hair and several layers of textiles, including a tunic with purple-dyed *clavi*, were found with adult and traces of Egyptian blue (calcium copper silicate known as *caeruleum*) with the child. As with the face of the adult from Naintré, exposed areas of skin appeared to have been coated with a layer of clay whose iron content provided a pink colouration. This supports details from the primary sources for the use of a fine powder to cover the progressive discolouration of the corpse (Erker 2011; Graham 2011; Hope 2009: 71). Plaster, gypsum for the adult and lime plaster for the child, was also present and, although no grave goods were extant, one of the lead coffins was engraved with a *chi-rho* symbol indicating that the individual was a Christian. Organic residue samples again provided evidence for resinous substances and demonstrated that the child had been interred with *Pistacia* spp. resin (mastic/terebinth) and the adult with a combination of *Pistacia* spp., conifer (Pinaceae, possibly pine) and *Boswellia* spp. (frankincense) exudates.

The most exciting finds, however, come from a number of exceptionally well-preserved sarcophagus burials from Trier, Germany which was one of the largest cities of the late Roman Empire and the capital of *Belgica Prima*. Dated to the 4th century AD, many of the individuals interred were members of the senatorial elite and/or officers at the court of the Emperor Constantine while some, at least, were Christians according to their epitaphs (Reifarth 2009). Studied as part of her PhD by Nicole Reifarth (2013, whose focus was the textiles), collaboration with Prof. Rainer Drewello, Bamburg, Germany and Prof. Carl Heron, University of Bradford, UK provided invaluable insights into other aspects of the treatment of the body. In addition to the results reported in detail regarding the evaluation of twenty-one sarcophagi (including one double burial and the inhumation of an adult with two infants) from the 'coemeterialbasilika' of St Maximin and a child burial from the abbey of St Matthias, Trier, samples from the grave of St Paulinus (also in Trier although he died in Phrygia, modern Turkey), residues from a glass vessel (Grave 53, St Matthias, Trier), a sarcophagus burial from Iovia, Hungary and another from the tower tomb of Atenatan, Palmyra were evaluated. Likewise, a fragment of textile from Burial 530, Poundbury, Dorchester, UK was investigated (Reifarth 2013: 460).

With regards to those from Trier, nine had been encased in gypsum and ten surrounded by wood shavings identified as fir (*Abies* spp.). Fir shavings were also identified in the tomb of St Paulinus while two burials of young females from St Maximin's were accompanied by twigs of myrtle (Reifarth 2013: 27-42). Although the skeletal remains were generally in poor condition all ages and both sexes were represented with hair remaining in a number of these sarcophagi, two of which had inner lead-liners. All of these individuals had been dressed and wrapped in a range of textiles (Reifarth 2013: 47-90). Analysis showed the presence of silk tunics often dyed with madder and adorned with decorative elements including 'Tyrian' purple-dyed wool *clavi* and/or stripes and floral motifs picked out using gold threads. Under-tunics of wool and fine gold threads (possibly woven into a linen fabric) were also identified with silk or bast fibre (flax?) shrouds over these garments and, on occasion, face covers of silk damask or pleated linen. In addition, two individuals had been carefully bandaged with strips of silk damask prior to being dressed while iron-containing pigments were found in association with two infants in the triple burial (Grab 174) and the post-cranial remains of a young adult (Grab 196) (Reifarth 2013: 112-113).

Figure 3.3. Resin-impregnated textiles, sarcophagus burial, Grab 571, St Maximin's, Trier, Germany (Image provided by Dr Nicole Reifarth, © N. Reifarth).

Finally, visible organic residues were present in all of the burials from Trier and appeared to have been poured over, incorporated within or pasted onto the vast array of textiles (**Figure 3.3**). Analysis indicated the presence of complex mixtures of oils/fats accompanied in many cases by gums and resins and/or gum-resins. Those confirmed by GC-MS consisted of conifer (Pinaceae and Cupressaceae) and *Pistacia* spp. resins with indications of a possible balsamic exudate (Reifarth 2013: 91-114). Evaluation of the samples from the tomb of St Paulinus (*Pistacia* spp. and a balsamic resin?) and the glass vessel from St Matthias (balsamic resin?) served to demonstrate the broad use of exotic aromatics in Trier. Likewise, investigation of burials from the Roman cemetery at Iovia, Hungary showed that the deceased had been tightly wrapped with linen bandages an dressed in silk, wool and gold decorated textiles over which perfumed substances had been poured. Evidence of pine and frankincense was provided in an earlier study (Sipos 2003 cited in Reifarth 2013) with Pinaceae (pine?) and *Pistacia* spp. resins from a double burial revealed by GC-MS (Reifarth 2013: 503). A similar pattern was discerned in the sarcophagus burial from the tower tomb of Atenatan, Palmyra which revealed that the body may have been desiccated and then bandaged with coarse linen, wrapped in fine linen or wool fabric and covered in silks with strips of richly decorated wool or silk fabric around the face, wrists and ankles before being enshrouded in a large linen cloth. These textiles were again impregnated with aromatic substances which have yet to be securely identified (Heron 2011, pers. comm., October; Reifarth 2013: 507-508; **7.5**).

Evaluation of this corpus of antiquarian and more recent research gives rise to a number of points of interest. The first is the issue of terminology. Where ‘mummified’ or ‘embalmed’ bodies are mentioned, it appears that observers may be referring to soft tissue preservation without distinguishing between natural and artificial mummification. For example, the dark residues described as resembling ‘leather’ noted in sandstone sarcophagi from Trier (grave catalogue cited by Reifarth 2013: 492) could derive from a number of sources. The presence of plaster and/or textile bandages and wrappings also appears, on occasion, to have been interpreted as evidence of ‘embalming’ (e.g. the ‘desiccated’ remains from Pannonia cited in Chioffi 1998: 71-73) even though these treatments do not necessarily imply the additional use of perfumed unguents. In addition, as Reifarth’s study has confirmed, “the chemical composition of ancient resins [rarely] correlates with their microscopic [let alone macroscopic] appearance” (2013: 126), and so colour and texture are not “of...diagnostic significance” (Mills and White 1989: 40). Thus, even in the case of visible organic residues, only when some form of chemical analysis has been undertaken can there be any surety about the inclusion and source of resinous plant exudates. Only a tiny fraction of the vast number of the Roman dead can, therefore, currently be shown to have received this form of body treatment.

3.4 Summary

Both the literary and archaeological evidence reviewed above supports the contention that aromatic plant products played an important role in the treatment of the dead. What is even more striking, is the extent of the commonalities between the burials found to contain resinous substances and their correspondence with the descriptions of the mortuary rites of the Roman elite reviewed in **Chapter 2**: "*There, crowded together in a long train, flow the spring produce of Arabia and Cilicia...her body lies on a high bier, veiled by silk, and Tyrian purple"* (*Statius Silvae 5.1:210-215* nd*).* Thus, with the exception of the multiple inhumations in the catacombs of *SS. Marcellino e Pietro,* Rome (although these also have related elements), all of these individuals had been placed in stone sarcophagi, sometimes with inner lead coffins and were, generally, accompanied by a variety of high quality grave goods. In addition, where taphonomic conditions have allowed, a range of fabrics including wool and damask silk, often with decorative elements provided by murex purple-dyed *clavi* (stripes) and gold tapestry-work, have been recovered (Bédat *et al.* 2005; Mitschke and gen. Schieck 2012; Wild 2013). This supports the hypothesis that both the use of and access to these costly materials, including an abundance of plant exudates, was correlated with the social status and wealth of the deceased. Nonetheless, as noted above, the better protection afforded by these substantial containers remains an issue in terms of taphonomic factors while organic residue analysis does not appear to have been undertaken on more normative burials.

Other elements such as the inclusion of wood shavings, the presence of plaster (lime or gypsum) and the application of pigments to the skin are also of considerable interest. Only the latter is mentioned in the literary sources while the use of fir bark appears, on current evidence, to be restricted to Trier despite the fact that plant materials in the form of twigs, 'herbs', crowns or garlands of leaves and flowers are frequently reported elsewhere (**Table 3.2; Table 3.3**). The use of plaster has, however, been more widely observed and appears to be a late Roman phenomenon whose significance has been much debated (Ramm 1971; Sparey Green 1977). All of these components seem to signify an increased attempt to disguise the visual and olfactory impact of decay and both their presence and properties are now becoming better understood as the result of focussed research agendas (e.g. Corbineau 2014; Devièse 2008; Reifarth 2013; Schotsmans 2013). The ritual and/or symbolic connotations of these substances must, likewise, have played a part as the ascription of meaning to both plant species and sensory effects such as colour in Roman eschatology are well attested (Bradley 2011; Giesecke 2014; Thomas 1979).

With regards to the plant exudates identified, both sources of evidence suggest that a restricted range of substances were used in the mortuary sphere. While the literature names a variety of scented materials, chemical analysis has provided definitive data for conifer resins (Pinaceae and Cupressaceae), mastic/terebinth (*Pistacia* spp.) and frankincense (*Boswellia* spp.). Traces of cinnamic acid and related compounds, sometimes in conjunction with triterpenic compounds, also indicates the presence of a balsamic resin such as *Liquidambar orientalis* and/or an essential oil (e.g. derived from cinnamon/cassia). There is, however, little direct correspondence between the primary sources and the archaeological evidence with conifer and *Pistacia* spp. resins, in particular, seeming to have played a significant, but unmentioned, role in the mid-late Empire. Nonetheless, the analytical work has confirmed that resinous exudates were, indeed, employed in the preparation of the deceased outside the confines of Rome and the eastern empire. All of the samples evaluated were, however, collected based on visual observations regarding the presence of organic residues. The possibility that invisible molecular evidence might remain within comminuted grave deposits protected by the burial environment afforded by stone sarcophagi and/or lead-lined coffins has not, therefore, been addressed. This has previously limited investigations into the treatment of the body in less favourable circumstances such as those encountered in Britain.

Chapter 4

Roman Britain: death and burial

"*There has been no systematic search for continental parallels for* [Roman] *burial practices...in Britain, an approach which is certain to yield valuable insights into the source of* [these] *customs*". Burial practices in Roman Britain, Philpott 1991: 2

4.1 Introduction

The most remote, north-western outpost of the Empire, the small island of *Britannia* was viewed as "another world" (Potter and Johns 1992: 12) even by educated Roman authors writing in the 3rd century AD. Due to this real and perceived distance from the centre of things in Italy, it has often been argued that the impact of the Roman way of life was more superficial in Britain than elsewhere in the Empire (Mattingly 2006: 528). Indeed, much of the history of the province can be characterised by conflict and military occupation due to the long-drawn out and only ever partial conquest, frequent rebellions, involvement in political schisms and the upheaval attendant on the final withdrawal of troops (Southern 2013: passim). The end of Roman rule also appears to have been followed by changes not widely observed in continental Europe denoted by the abandonment of the trappings of Rome including urban living, construction in stone, use of coinage and, ultimately, by language change with the coming of the Anglo-Saxons (e.g. Hills 2003; Reece 1980; Salway 1981: 413-501). Thus, despite the overarching 'Romanization' of Britain in terms of planned towns, bathhouses, villa complexes, mosaics and even food-stuffs (e.g. garum), the depth of cultural change is hard to ascertain. We know little about the extent to which the minutiae of Roman customs were adopted (Mattingly 2006: 3-22).

With regards to the current research agenda, this view is supported by a lack of funerary monuments, the minimal use of epigraphy (resulting in few tomb inscriptions) and scant evidence for widespread conversion to Christianity (Thomas 1981: 230-239; Watts 2011: 121-137). There are, for example, no *arcosolia*, *columbaria*, catacombs or clear evidence of streets of tombs and no upstanding mausolea, burial vaults or crypts below subsequent Christian churches such as those on the continent that have provided evidence for elaborate body treatments (**3.3**). In addition, during the period when most examples of the rite of interest have been identified (late 2nd-4th centuries AD), the importation of luxury goods to Britain appears to have been in decline (Mattingly 2006: 513-520). The possibility that costly, unfamiliar substances may have been introduced into the notoriously conservative sphere of mortuary traditions and regularly employed in burial contexts across the province has, therefore, largely been dismissed. Indeed, at the start of this project, even the author was highly sceptical.

Nonetheless, a renewed interest in Roman studies combined with the application of mortuary theory has served to increase our understanding of the range and complexity of mid-late period burial rites in Britain. A complete chronicle of this evidence is neither possible nor desirable as previous research has established the key features of Roman practices (e.g. Pearce 2013; Philpott 1991; Salway 1981: 693-707). Thus, only a brief overview of pre-Roman Iron Age (PRIA) mortuary traditions (**4.2**) and consideration of the impact of Rome on the subsequent trajectory of funerary practices (**4.3**) is offered here in order to place the rite of interest in context (**4.4**) and provide a list of targets for sampling as summarised in **4.5**.

4.2 Pre-Roman Iron Age (PRIA)

Classical sources are largely silent about the mortuary rites of the Celtic peoples with only the comments of Caesar as evidence for practices in Gaul: *"their funerals...are splendid and costly...everything that the dead man [was]...fond of...is placed upon his pyre"* (*Caesar 6.19:4* nd). How the ashes were subsequently treated is not specified. This poses a particular problem when considering PRIA Britain as the vast majority of the dead were disposed of in an archaeologically invisible manner (Whimster 1981: 267-268; Wait 1985: 236). Those skeletal remains that have been recovered largely comprise disarticulated and fragmentary body parts which appear to represent the selective retrieval of skeletal elements from exposure grounds and/or after curation within settlements (Carr and Knüsel 1997; Craig *et al.* 2005). In addition, indications for the continuance of cremation, the dominant rite in the Bronze Age, have recently come to light in specific contexts (e.g. placed within cists in southern Scotland, Armit *et al.* 2013). Some of these finds pertain to the 6th century BC but most are later in date and seem to reflect a common tradition that extended across much of the British Isles in the PRIA (Esmonde Cleary 1992: 29-30). The nature of the normative rite during this period has, therefore, been the focus of considerable debate with various explanations proffered from cremation and the scattering of ashes, to disposal in water and cannibalism (Armit and Ginn 2007).

Current evidence favours the practice of excarnation accompanied by a range of minority rites involving the infrequent deposition of complete articulated skeletons, generally crouched or flexed, as isolated inhumations and the peri/post-mortem processing of the remains of a small percentage of the population (Armit *et al.* 2013; Madgwick 2008; Wait 1985: 237-240). This is supported by osteological evidence for cut marks indicative of disarticulation and defleshing, dry fractures and canid gnawing demonstrating exposure and occasional examples of bone modification denoted by drilled holes and inscribed decoration (Redfern 2008a; Tucker 2010: 295-315). Thus, PRIA human remains in various states of (dis)articulation are regularly recovered from pits, middens, boundary ditches, beneath floor surfaces and the ramparts of hillforts and even within the walls of dwellings (Armit and Ginn 2007; Tucker 2010: 129-220). Although disturbance of articulated individuals must have occurred, most of these finds appear to form part of deliberate deposits often related to entrance ways and the foundation or abandonment of structures (Armit and Ginn 2007; Mattingly 2006: 477). This interpretation is supported by the frequent recovery of ceramics and other artefacts from these contexts alongside portions or whole skeletons of other species (e.g. pigs, dogs, horses) (Bendrey *et al.* 2010; Redfern 2008a). Despite being buried together, differential treatment of the human remains is evident as only other animals appear to have been butchered, cooked and consumed (Armit and Ginn 2007; Madgwick 2008).

There is also a strong argument for the selection of specific skeletal elements with a “disproportionate occurrence of [human] heads” (Armit and Ginn 2007:

127). Predominantly male and commonly defleshed (Mattingly 2006: 477), this phenomenon has been identified in both Britain and continental Europe and appears to reflect a culture of head-hunting and head-veneration among the 'Celtic' peoples (Armit 2006). Indeed, observations by classical authors include an interesting comment about the method of preservation employed in certain circumstances: "*the heads of their most distinguished enemies they embalm in cedar-oil...preserve in a chest, and...exhibit to strangers*" (*Diodorus Siculus 5.29:5* nd). Not all of these severed heads may have been trophies, however, as special treatment of the skulls of ancestors or recently deceased group members may have occurred as part of "conventional funerary rites" (Armit 2006: 11).

Within these overarching patterns, a number of regional variants start to appear between the 4th-1st centuries BC. These provide a more substantial archaeological record as a percentage of the population began to be interred in a formal manner (Esmonde Cleary 1992). Most of these geographically restricted traditions seem to be insular in origin and are characterised by crouched or flexed inhumations (generally aligned NS/SN) although extended inhumations in flat-graves within small defined burial grounds or as family groups are observed in all areas (Watts 2011: 122; Whimster 1981: 275). Thus, in the south-west a series of cist-graves, some containing metalwork, have been identified, while in the area of the Durotriges (Dorset) a distinctive rite comprising simple earth-cut inhumations accompanied by a restricted range of grave goods (e.g. ceramics, jewellery, food offerings) was established by the 1st century BC and continued into the Roman period. In eastern Yorkshire, individuals supplied with a similar range of artefacts were interred in rock or gravel-cut graves below small square barrow enclosures with a later sub-group of extended, EW orientated burials found around Burton Fleming. Referred to as the Arras culture due to possible links with continental practices, this tradition is best known for its relatively extensive cemeteries and more elaborate manifestations as certain individuals were interred with wheeled vehicles (Whimster 1981: 93-158).

Finally, in the south-east, urned cremations in flat-graves accompanied by both burnt and unburnt grave goods appear during the 1st century BC (Esmonde Cleary 1992; Whimster 1981: 269-275) with more elaborate, composite rites observed at a number of sites (e.g. St Alban's, Stanway, Baldock, Lexden, Westhampnett). The latter are characterised by cremated remains placed within central chambers or shafts below ditch-enclosed barrows denoting 'Celtic' customs with the status of the deceased further materialised through an array of imported artefacts (e.g. Roman-style military chain mail, horse trappings, wine amphorae and other ceramic vessels, metal wares ranging from statuettes to gold threads and parts of ceremonial chair and stool). These southern and eastern burial rites, which appear to have been influenced by contact with Belgic Gaul, extended into the early post-Conquest period and may have signified client-king or military-service relations with the Roman Empire (Black 1986; Mattingly 2006: 77-80, 477-478). Likewise, a diffuse rite of inhumation with weaponry (e.g. swords, spears, knives) discussed by Whimster (1981: 159-187), again indicates an emphasis on social differentiation as denoted by imported grave goods.

What do these "fluid and pluralistic" (Armit *et al.* 2013: 96-97) practices tell us about attitudes towards the body in the PRIA? One aspect appears to be a belief in the maintenance and manipulation of relationships between the living and the dead through continued interactions with specific skeletal elements or the deposition of articulated individuals within domestic contexts (Madgwick 2008). Both curation and display of human remains is indicated with the treatment of the deceased seemingly related to their role in life based on ethnographic analogies (e.g. as outsiders/enemies, members of the elite, great warriors/shaman) (Armit *et al.* 2013; Wait 1985: 235-245). Support for this hypothesis comes from the contrast between an abundance of peri-mortem injuries indicative of interpersonal violence on disarticulated elements or articulated isolated burials and minimal indicators of trauma on the skeletal remains of those interred in burial grounds (Armit *et al.* 2013; King 2010: 235-264). These findings have been interpreted as the use of ritual to re-establish stability and mediate social tension either in the face of sudden death (e.g. raiding/warfare) or through the selection of suitable sacrifices (King 2010: 235-264). Thus, for certain individuals the normative rite, probably excarnation, was interrupted to enable them to act in diverse ways as ritual agents within a culture that revered the power of the human body (Armit and Ginn 2007: 129; Tucker 2010: 316-342).

4.3 Roman period mortuary practices

During the approximately 350 years of Roman rule mortuary practices in Britain, although "dynamic and regionally varied" (Philpott 1991:1), appear to have largely followed the course traced elsewhere in continental Europe (Mattingly 2006: 343-347). Thus, despite an absence of references in the contemporary literature regarding post-conquest burial rites, changes in the method of body disposal have facilitated archaeological investigations (Esmonde Cleary 1992). The most significant being that, with the arrival of Rome, a far larger percentage of the population became visible in death due to the requirement under Roman law for formal interment in extra-mural burial grounds (Jones 1987). Most evident in the south-east and around urban sites, an increase in the range of imported goods (e.g. Samian wares) deposited in these graves has also been observed which, together with the introduction of epigraphs, attest to influences and immigration from Gaul in particular (Esmonde Cleary 2000; Philpott 1991: 217-218).

Likewise, the spread of urned (or otherwise contained) cremation burial shows that novel practices were adopted, although at a relatively slow and varied pace, with some regions maintaining their PRIA traditions (Pearce 2000). For example, in the area of the Durotriges, crouched inhumation continued until supplanted in the 2nd-3rd centuries AD by extended inhumation (Esmonde Cleary 1992). Nonetheless, as initiated in the late PRIA, materialised social differentiation is increasingly evident, denoted by more elaborate containers (e.g. glass vessel, casket and/or box burials), composite rites (e.g. under a barrow) and imported grave goods, again reflecting links with the Gallic provinces (Mattingly 2006: 77-80; Philpott 1991: 220-221). In northern and western Britain, however, as well as in more rural regions, far less evidence of formal cremation or inhumation burial has been recovered. This may be an artefact of excavation bias (Esmonde Cleary 1992). The archaeological invisibility of the majority of PRIA rites, however, makes their retention due to conservatism or opposition difficult to ascertain especially as disarticulated body parts and burials in boundary features have been regularly identified in post-Roman contexts (Mattingly 2006: 478).

It was not, therefore, until the 3rd century AD that the normative Roman rite, which by this period was extended, EW orientated, inhumation in a shroud and/or wooden coffin, appears to have been more extensively adopted across the province (Philpott 1991: 222-228). Once established, subsequent trends continue to mirror those observed elsewhere in the Empire with a more standardised package of grave goods (where present), a reduction in the furnishing of graves and the development of more ordered burial grounds around large urban centres in the 4th century AD (Jones 1987; Rahtz 1977). Thus, a trajectory towards "an increased concern for [maintaining]...the physical integrity of the body" is indicated during the mid-late Roman period (Philpott 1991: 238). The extent to which these modifications in mortuary practices reflect changes in religious affiliation are impossible to determine with the influence of Christianity, in particular, much debated (e.g. Philpott 1991: 235-240; Thomas 1981: 227). This is complicated by the fact that very little is known of PRIA beliefs regarding the afterlife (Wait 1985). The inclusion of food-stuffs, ceramics, jewellery and weaponry alongside the widespread adoption of footwear, lamps and coins as grave goods suggests, however, that concepts relating to a journey to a continued existence were prevalent from the late PRIA to the end of the Roman period (Black 1986).

Nonetheless, with the advent of widespread inhumation, although cremation persisted as a minority rite throughout the Roman period (Mattingly 2006: 343), more complex questions about the burial population become easier to address. Of potential relevance to this study (i.e. in order to compare individuals interred with resinous substances to the general population), osteological and palaeopathological research has, in regions where comparison with the PRIA can be made (e.g. Dorset), indicated low life expectancy and an increased prevalence of environmental stress, certain diseases (e.g. tuberculosis, rickets, scurvy) and levels of violence (Bonsall 2013; Cool 2006: 21-29; Morris 1992: 89; Redfern 2008b). Isotope studies have provided additional evidence for the consumption of a predominantly terrestrial diet and considerable population mobility (e.g. Eckardt *et al.* 2009; Evans *et al.* 2006; Montgomery *et al.* 2010; Redfern *et al.* 2012). An image of Roman Britain as a melting pot of immigrants (often of relatively high social status), widely-travelled provincials and stay-at-home locals with discrepant experiences of Imperial rule is, therefore, being steadily revealed (Eckardt *et al.* 2014; Mattingly 2006: 491-528).

Variants in the treatment of the body can also be discerned and need to be placed within this broader context. For example, the move from NS to EW orientation appears to be externally-influenced and fairly generalised whereas specific rites (e.g. decapitation) may reflect the continued influence of native traditions (Armit *et al.* 2011; Mattingly 2006: 346-347; Philpott 1991: 77-89). Other forms of disposal (e.g. the differential treatment of infants) appear, however, to indicate congruence between aspects of pre-Roman and Roman practices (Armit and Ginn 2007; Mattingly 2006: 479; Millett and Gowland 2015). One notable similarity seems to have been the element of display with conspicuous consumption employed to signify the social status and/or wealth of the deceased both before and after the Conquest (Philpott 1991: 219). Expressed through the agency of materials, in the Roman period this aspect of identity appears to have been reflected in the method of containment as well as through the artefacts and substances selected to accompany the deceased to the pyre or tomb (**2.3; 4.2**). This brings us to the rite of interest, the potential importation of resinous plant exudates for use in the treatment of the body as part of the suite of Roman mortuary practices introduced to Britain.

4.4 The rite of interest

Antiquarian reports from Britain furnish a limited number of references to the presence of unusual residues or body preservation in Roman mortuary contexts (**Table 4.1**). Most of these reports describe materials (solids and/or liquids) associated with grave goods accompanying cremation burials or found within the cremation vessel itself. Gage (1834; 1836), for example, mentions a number of accounts regarding liquids in cremation urns from both Britain (e.g. Southfleet, Kent; Winston, Derbyshire) and continental Europe (Italy and France). Some of these aqueous fluids were reportedly mixed with organic, “oleaginous” (Litlington, Hertfordshire; Gage 1836: 19) or “unctuous” (Withersfield, Cambridgeshire; Gage 1936: 314), matter. In addition, white substances on the inner surface of basketwork in the shape of a bottle (from a barrow at Bartlow Hills) and on a Samian ware dish (alongside some leaves, with a cremation *amphora*, Weston Turville) appeared, upon analysis in the 1800s, to be gum-resins (Gage 1834; Waugh 1962). These observations suggest that exotic resinous substances may have reached Britain in the early Roman period and were deemed acceptable for inclusion in mortuary contexts as part of a suite of unburnt offerings.

The only hints, however, that some form of body treatment involving aromatic preservatives might have been introduced into the province come from antiquarian descriptions of two sarcophagus burials from Dartford, Kent (Dunkin 1844) and two more recent finds. The latter comprise a brief reference to the remains of a child (c. eight years old) interred in a sarcophagus near Glaston, Leicestershire (formerly Rutlandshire) having “exuded a pleasant odour ‘like scented disinfectant’” (Webster 1950: 72) and more substantial evidence, found with an infant (c. 11-12 months old), discovered near Arrington, Cambridgeshire (Taylor 1993). Inhumed in a lead-lined coffin which was excavated under laboratory conditions, “numerous pieces of aromatic resin, still smelling distinctly of incense” (Taylor 1993: 194) were recovered from around the cranium. The resinous nature of these fragments, but not their botanical source, was confirmed by chemical analysis. As with the Dartford discoveries, traces of plaster were present with this infant from Arrington which sparked new interest in a class of late Roman burial involving plaster body-casings proposed by Ramm (1971) whose ideas were subsequently developed by Sparey Green (1977; 1982, *et al.* 1982).

Table 4.1. Previously reported instances of organic, potentially resinous, residues in Roman period mortuary contexts from Britain.

Location ***Found/Reference***	***Burial type*** **Date (AD)**	**Age/sex**	**Container**	**Associated materials**	**Organic residues**	**Current location**
Dartford, Kent *Discovered c. 1797* *Dunkin 1844*	*Inhumation* Late Roman	Adult	Stone sarcophagus	Plaster retaining textile impressions.	Unusually well preserved; dark appearance to skin; "fell to dust" when touched.	Human remains reburied soon after discovery
Dartford, Kent *Discovered c. 1822* *Dunkin 1844*	*Inhumation* 3rd-4th century	Adult Female	Stone sarcophagus	Hair present; 'bandeau' of pearls; coin; textiles; plaster.	Organic residue which retained an aromatic smell, adhering to larger skeletal elements.	Human remains reburied in 1822
Bartlow Hills Essex/Cambridgeshire *Gage 1834*	*Cremation* Barrow 2 Early Roman		Green glass vessel in tile chamber below barrow	Gold intaglio ring; coin of Hadrian; glass vessels; basketwork 'bottle'; wooden box with iron fittings.	Analysed by M. Faraday (1832): aqueous liquids in urn and glass vessel; fatty material in bottle; white substance, ?gum-resin, on basketwork.	Relevant materials destroyed in a fire
Bartlow Hills Essex/Cambridgeshire *Gage 1836*	*Cremation* Barrow A Early Roman	Adult	Green glass urn in wooden box below barrow	Bronze items e.g. a patera, enamelled vessel, lamp, jugs, strigils; glass and ceramic vessels; folding stool.	Analysed by M. Faraday: combustible solid substance in lamp; black solid (?asphalt) on neck of bottle; sugar/aqueous mixture and fatty matter (?honey and oil) within bottle.	Relevant materials destroyed in a fire
Weston Turville Buckinghamshire *Discovered 1855* *Waugh 1962*	*Cremation* Mid-late 2nd		Clay amphora	Glass and ceramic (including Samian) vessels; brooches; bone pin; beads; hobnails; mirror (frags); ?wooden box.	Some leaves and a white substance which emitted an aromatic scent were found on a Samian-ware dish. Analysis (in 1800s) indicated the substance was frankincense.	Human remains reburied; no residues extant (2014)
Glaston Leicestershire *Discovered 1947* *Webster 1950*	*Inhumation* Late Roman	Child c. 8 years old	Stone sarcophagus	Flakes of iron; fragments of 2 glass vessels; 4 small bronze bracelets.	Fragments of bone described as giving off a pleasant odour similar to scented disinfectant.	Unable to be located by museum staff (2013-2014)
Dorchester, Dorset Poundbury *Farwell and Molleson 1993* *Sparey Green 1977, 1982*	*Inhumations* 3rd-4th century	All ages Both sexes	Stone sarcophagi and/or lead-lined coffins; some below mausolea	Many containing plaster and textiles; gold thread (B530); some hair present; minimal grave goods e.g. combs, coins	Organic residues noted in a number of these burials often in association with textiles e.g. Burial 530.	Extant; curated by DNHAS/ DMT
Dorchester, Dorset Crown Buildings *Sparey Green et al. 1982*	*Inhumation* ?4th century	Adult	?Two lead-lined wooden coffins; one excavated; one badly damaged	The one recovered contained gypsum (partial fill); textile impressions; 'head' and plait of human hair; no grave goods	Dark matter observed adhering to the human hair.	Extant; curated by DNHAS/ DMT
Arrington, Cambridgeshire *Taylor 1993*	*Inhumation* 2nd c. or later	Infant c. 1 year old	Lead-lined oak coffin	Plaster; fragments of dyed wool; hair fragments (light brown); pipeclay figurines possibly in a wooden box.	Numerous brittle, yellow-brown fragments around the cranium, still odorous. Initial chemical analysis suggested a plant resin.	Extant; curated in MAA, Cambridge

This form of body treatment was described by Toynbee (1996: 41) in relation to burials around York, North Yorkshire, UK as “not infrequently gypsum [i.e. plaster, see below] was poured over the body, forming a cast...and sometimes preserving fragments of the textile in which it had been wrapped”. Listed in the Royal Commission on Historical Monuments of England (RCHME) volume, *Eburacum* (1962), these finds were shown by Ramm (1971) to form a sub-set of the 3rd-4th century AD inhumations from York with other examples from Yorkshire and southern Britain also noted. The vast majority of those identified were recovered from stone sarcophagi and/or lead-lined coffins with traces of textiles (shrouds and/or garments) indicated by impressions on the plaster and/or fragments of mineral-replaced cloth. Based on similar discoveries in North Africa (e.g. Algeria) and the Rhineland (e.g. Trier) where earlier or contemporary examples of plaster body-casings, coatings and “beds of lime” (Ramm 1971: 189) had been reported, he argued that this practice represented an imported ‘package’ possibly linked with the spread of Christianity. Since *Eburacum* (like Trier) became the seat of the imperial court twice during this period (under Septimius Severus, 193-211 AD and Constantius Chlorus, 293-306 AD) it was also viewed as a “fashionable and expensive” body treatment (Ramm 1971: 197) which formed part of a range of options available to a “cosmopolitan entourage whose diverse origins...encompassed a range of burial traditions” (Philpott 1991: 224).

Subsequently, interest in the use of plaster continued to grow as details of many such body-casings in burials from around Dorchester were published, most notably those from the main burial area at Poundbury (Davies and Grieve 1987; Sparey Green 1977, 1982, *et al.* 1982; Woodward 1993: 227-228). Thus, by the early 1990s Philpott (1991: 90-96, 310-313) was able to compile a list of over 130 plaster burials, including some in wooden coffins, with additional examples described elsewhere (e.g. Barber and Bowsher 2000: 101-104), some of which remain unpublished (e.g. Fulford, Mcnab 1997). Most of those identified pertain to burial grounds serving large urban sites (e.g. London, York, Dorchester) although discoveries at villa complexes (e.g. Lullingstone, Kent) and in other rural locations have been reported (Philpott 1991: 90). In addition, evidence from Colchester (G687, a plaster burial with a ceramic vessel coated internally with a thick layer of lime) and the eastern burial ground of London (pits containing traces of chalk slurry) support the contention that plaster, when present, was added at the graveside (Pinter-Bellows 1993: 36-37; Whytehead 1986).

Chemical analysis of a small number of these finds demonstrated that both calcium carbonate-based substances (e.g. lime and chalk) and calcium sulphate (gypsum) could be used in this manner (Barber and Bowsher 2000: 101-103; Pinter-Bellows 1993: 36-37, 126; Sparey Green *et al. 1*982; Whytehead 1986). Nonetheless, as most of the white substances reported were not assessed, misidentifications abound in the literature, as highlighted in the doctoral thesis of Eline Schotsmans (2013). This study has shown that white solids or tide-marks in mortuary contexts may derive from a range of intrinsic (e.g. degraded bone, brushite crystals, adipocere) or extrinsic sources (ingress from the burial environment due to dissolution in water or via cracks in the burial container) (Schotsmans *et al.* 2014a). Thus, although full or partial body-casings can be interpreted as deliberate depositions, more ephemeral traces require careful consideration before identifying plaster burials (e.g. at Alington Avenue, Dorset, **7.8**). Which material was applied also cannot be distinguished without analysis especially as both gypsum and lime were used, sometimes within the same burial ground (e.g. Poundbury, Dorset, **7.8**). In addition, this research confirmed that plaster, regardless of type (lime or gypsum) or method of application (hydrated or powdered), forms a cast which acts as a temporary preservative (for at least 6 months) by containing putrefactive odours, absorbing body fluids and delaying invasion of the corpse by decomposer organisms and scavengers (Schotsmans *et al.* 2012). Ultimately, however, degradation resumes and skeletalisation occurs (Schotsmans *et al.* 2014b, 2014c), a fact realised by Ramm (1971), although often the source of much confusion elsewhere in the literature.

The practical aspect of this late Roman body treatment has, therefore, now been clarified although this does not preclude a ritual meaning. Indeed, the significance of plaster has been much debated and has become embroiled in the controversy over the identification of Christians in the burial record (Philpott 1991: 93-95). Originally suggested by Ramm (1971), salient features of these arguments are that the use of plaster, specifically gypsum, originated in northern Africa where it was adopted by Christian communities. It then spread due to the cosmopolitan nature of the Empire and a correspondence between attempts to preserve the body and the doctrine of physical resurrection. This relationship has been strongly advocated by Sparey Green (1977, 1982) with plaster subsequently cited as a marker of Christian burial (Woodward 1993: 236-237). Part of a desire among some western researchers to prove the widespread presence of Christians in Roman Britain (e.g. Petts 1998; Sparey Green 2003, 2004; Watts 1991: passim), many flaws have been highlighted in this reasoning (Philpott 1991: 239-240; Thomas 1981: 230-239). Alternative explanations ranging from the influence of other religious cults to associations with status and wealth have, therefore, been posited (Black 1986; Morris 1986; Toynbee 1996: 42).

Of these, it is the association with status that provides the strongest case (Philpott 1991: 228-233), particularly in light of the importance placed on this aspect of identity in the Roman mortuary sphere (**2.3**). This correlation with social standing was also proposed by Ramm (1971) and considered by Sparey Green (1982). An apparent hierarchy within the category of plaster burial may support this contention as more substantial body-casings seem to be linked with interment in sarcophagi and lead-lined coffins. Taphonomic issues around the better protection afforded by these substantial containers, however, creates a significant flaw in this argument due to the greater potential for leaching from more normative inhumation burials. Nonetheless, analysis of samples from Poundbury, Dorset indicates that gypsum (less easily obtained than lime), was present only when these 'package' burials were additionally placed within mausolea (Brettell *et al.* 2015a; **7.8**). Likewise, at Butt Road, Colchester, although a layer of lime plaster was present in a number of wooden coffin burials only one individual had been encased in gypsum, a child c. two years old interred in a lead-lined coffin (Pinter-Bellows 1993: 126). Thus, this apparent exclusivity of gypsum, rather than being linked to a specific set of religious beliefs, may be related to its relative scarcity in comparison with calcium carbonate and its appearance as it is more reflective/whiter than lime (Schotsmans 2012, pers. comm., March-November). Indeed, the colour white, alongside red, was closely linked with generic funerary symbolism in the Roman world (Thomas 1979).

The significance to the present study of this research into the use and meaning of plaster is twofold. Firstly, a relationship between this class of burial and ‘embalming’ has been mooted based on residues noted with one find from Dartford, the individual from Crown Buildings, Dorchester and during excavations at Poundbury (Philpott 1991: 92; Sparey Green *et al. 1*982; Woodward 1993: 228). This claim, although unsubstantiated, merits examination since the material components of this ‘package’ clearly represent an additional level of investment seemingly aimed at increased protection of the cadaver. In addition, a trajectory towards maintaining the integrity of the body can be traced throughout the late PRIA and Roman periods which may relate to belief in a more substantial afterlife (Edwards 2007: 218-220; Philpott 1991: 94). Secondly, Schotsmans’ PhD thesis (2013) has demonstrated the potential increase in knowledge and understanding of aspects of body treatment (both ancient and modern) as a result of detailed, contextualised, chemical analysis using modern instrumental techniques.

Thus, the discovery of visible resinous fragments from Arrington, Cambridgeshire in conjunction with the evidence building in continental Europe (**3.3**) provided the impetus for investigation of the use of plant exudates as part of mortuary rites with regards to Roman Britain. A list of plaster, stone sarcophagus and/or lead-lined coffin burials where skeletal remains and/or associated grave deposits were believed to be extant was, therefore, compiled (**Table 4.2**). These were selected as, based on current evidence (archaeological and literary, **Chapter 3**), they were considered more likely to have originally contained an abundance of resinous substances but also, due to the impact of taphonomic processes on less well protected inhumation burials, to provide the best chance of recovering chemical traces of this body treatment from British contexts. Recent discoveries identified during enquiries regarding access to samples and due to contacts made as the result of conference presentations are also included.

4.5 Summary

Comparison between PRIA and Roman mortuary practices in Britain demonstrates considerable differences in terms of the disposal of the dead. In the PRIA, across much of the British Isles, the normative rite was archaeologically invisible and may have involved excarnation. Only those whose status, possibly as venerated ancestors or defeated enemies, marked them out as special in some manner were selected for other forms of body treatment which served to maintain their links with the living. This included articulated burial in domestic areas, full or partial display (e.g. heads) and the manipulation of other elements (e.g. decoration and/or perforation perhaps for use as talismans) prior to secondary burial. In the late PRIA, a trajectory towards more formal burial rites appears in a number of areas, with links to the continent seemingly having influenced a move towards contained cremation in areas of the south and east of Britain (**4.2**).

The coming of Rome gave added impetus to this trend and denoted a marked change in body disposal practices for much larger swathes of the population as the conquest moved across Britain (**4.3**). Extensive cemeteries were established around the perimeters of urban settlements with smaller delineated burial grounds found in more rural regions. The '*mos Romanus'*, urned or otherwise contained cremation burial, was extended across much of the south and east of the province and was prevalent in militarised zones elsewhere while in other areas (e.g. Dorset) crouched inhumation continued but now within formally demarcated areas for the dead. In the later Roman period, mirroring changes elsewhere in the northern Empire, further standardisation in disposal rites can be traced. Thus, the practice of extended, coffined inhumation, often with minimal or no grave goods, was widely adopted within burial grounds that show signs of increased organisation (e.g. interred in rows, generally EW aligned).

Table 4.2. Mortuary contexts targeted for analysis (for full details see respective **Case Studies, Chapter 7**).

Location ***Reference***	***Burial type*** **Date (AD)**	**Age/sex**	**Container**	**Associated materials**	**Organic residues**	**Current location** **SAMPLED/not**
Arrington, Cambridgeshire *Taylor 1993*	*Inhumation* 2^{nd} c. or later	Infant c. 1 year old	Lead-lined oak coffin	Plaster; fragments of dyed wool; some hair; pipeclay figurines from Gaul, possibly in a wooden box on foot of coffin	Yellow fragments around cranium, still odorous. Initial analysis suggested a plant resin.	Extant; curated by MAA, Cambridge **SAMPLED 2011**
Cirencester, Bath Gate, Gloucestershire *Viner and Leech 1982*	*Inhumations* 3^{rd}-4^{th} century	All ages; both sexes; c. 9 month infant in lead-lined sarcophagus	25 stone sarcophagi, 1 lead-lined	Some with grave goods e.g. glass vessels, hobnails, chicken bones; others without grave goods	None reported	Some missing; others left in situ or in open air; lead-liner 'stolen'; **Not found/assessed**
Colchester, Essex *Crummy et al.1993*	*Inhumations* 3^{rd}-4^{th} century	All ages Both sexes	6 wood coffins c. 15 lead-lined coffins	Lime plaster; 1 with bowl from which plaster had been poured 1 with lime plaster; another (c. 2 yr old child) encased in gypsum Minimal/no grave goods; textiles	None reported	Curated in Colchester Museum **Closed for researcher access during PhD**
Dorchester, Dorset Poundbury *Farwell and Molleson 1993* *Sparey Green 1977*	*Inhumations* 3^{rd}-4^{th} century	All ages Both sexes	10/11 stone sarcophagi, 1 lead-lined; 26 lead-lined coffins; some below mausolea	Many containing plaster and evidence of textiles; gold thread (in 530); some hair present; minimal grave goods e.g. combs, coins	Organic residues noted often in association with textiles e.g. Burial 530	Extant; materials curated by DNHAS/ Dorset Museum Trust (DMT) **SAMPLED 2012**
Dorchester, Dorset Crown Buildings *Sparey Green et al.1982*	*Inhumation* ?4^{th} century	Adult	?2 lead-lined coffins; 1 excavated, ?possible denoted by fragments	1 recovered; gypsum (partial fill); textile impressions; 'head' and plait of human hair; no grave goods	Dark matter observed adhering to the hair	Extant; curated by DNHAS/ DMT **SAMPLED 2012**
Fordington, near Dorchester, Dorset Alington Avenue site *Davies et al. 2002*	*Inhumations* 3^{rd} century	Adult female Child c. 8 yrs old	Stone lid, wood coffin Lead-lined coffin	Reported as plaster burials (actually chalk ingress); murex-purple dyed stripes on tunic of child; hobnails, glass and ceramic vessels, coin	None reported	Extant, except infant; curated by DNHAS/ Dorset Museum Trust **SAMPLED 2012**
Icklingham, Suffolk *Prigg 1901:65-71 cited in* *Sparey Green 1977*	*Inhumations* 3^{rd}-4^{th} century	Adult males Adult female	2 stone sarcophagi 1 lead-lined coffin	1 encased in plaster	None reported	Possibly curated in Colchester Museum **Closed for access**
London, northern cemetery 'Spitalfields Lady', *Museum of London 1999* *Thomas 1999*	*Inhumation* 4^{th} century	Young adult female	Stone sarcophagus with inner lead coffin below timber mausoleum	Textiles (silk damask; wool; purple-dyed elements); gold threads; x8 glass vessels; bay leaves; glass phial; jet artefacts	None reported	Extant; curated by Museum of London (MoL) **SAMPLED 2012-2013**
London, various sites *Barber and Bowsher 2000* *Cowan 2003; Hall 1996* *Mackinder 2000*	*Inhumations* 3^{rd}-4^{th} century	All ages Both sexes	c. 15 stone sarcophagi; c. 14 lead-lined coffins; c. 90 wood coffins	c. 110 of these contained chalk or plaster; range of grave goods e.g. ceramic and glass vessels, coins, hairpins; others unaccompanied	None reported	Extant; curated by Museum of London **SAMPLED 2013-14**
Newcastle *Discovered 2008* *Unpublished*	*Inhumations* 3^{rd} century	Adult and child Poorly preserved	Two stone sarcophagi	Jet hairpin	Completely waterlogged	**Survival of relevant residues unlikely**

Purton, Wiltshire Northview Hospital site *Chandler 1994; McKinley 1994; Nurse 1992*	*Inhumations* 3rd- 4th century *Cremation* ?no date	3 adults ?All females – possibly related	2 stone sarcophagi; 1 glass vessel in lead urn in limestone ossuary	Textiles; ?plaster; coin; ceramic and glass vessels; shale bracelet; hobnails; animal bones	1 with dark residue on post-cranial elements. Fluid and organic matter (fat/oil) in glass urn.	Extant; curated by Swindon Museum and Art Gallery **SAMPLED 2014**
Salisbury, Wiltshire Boscombe Down site *Discovered 2007 McKinley 2013, pers. comm., 27 Mar.*	*Inhumation* 3rd century	Double burial Adult female c. 7 year old child	Stone sarcophagus	Leather shoes with cork soles/fur-lining (adult); calf skin footwear (infant); textiles; jet necklace; copper anklet; ceramic vessel	None reported; exceptional organic preservation	Extant; curated by Wessex Archaeology **SAMPLED 2013**
Somerset, various sites e.g. Northover House *Leach 1994*	Inhumations 4th century	Both sexes Adults	Stone sarcophagi and/or lead-lined coffins		None reported	Missing or reburied in situ; minimal details **Not extant**
Ilchester, Somerset *Discovered 2013 Minnett 2013, pers. comm. 08 Nov.*	Inhumation 2nd-3rd century	Young adult female	Lead-lined coffin	Hobnails (pair of shoes); decorated black (?jet) bead	Lead-liner damaged by plough etc; filled with soil; water/faunal ingress	Author assisted with excavation in lab. **SAMPLED 2013**
Winchester, Hampshire *Morris 1986; Richards 1999; Ottaway et al. 2012*	*Inhumations* 3rd- 4th century	Both sexes Adults	5 lead-lined coffins	Textile fragments; 1 with gold thread; range of grave goods e.g. coin; bone comb	None reported	2 extant; curated by Winchester Museums **SAMPLED 2012-13**
Witherly, Leicestershire *Discovered 2013 Keeley 2013, pers. comm., 13 Dec.*	*Inhumation* ?4th century	?infant; minimal bone extant	Lead-lined coffin	Items of jet	Full of clay; nothing present	**Examined by researchers at York**
York, North Yorkshire *RCHME (Eburacum) 1962 Ramm 1971; Toller 1977*	*Inhumations* 3rd- 4th century	Both sexes All ages	Over 50 stone sarcophagi and lead-lined coffins	Many containing plaster; evidence of textiles; range of grave goods	None reported; examined by N. Reifarth 2010, rejected as no resinous fragments visible.	Some sarcophagi and plaster extant; curated by York Museum Trust **SAMPLED 2014**
Hungate, York *Discovered 2011 Keeley 2013, pers. comm., 13 Dec.*	*Inhumation* 3rd-4th century	?	Lead-lined coffin	?	Frankincense probably present – awaiting full results from analysis.	**Being analysed by researchers at York**
Many other reported finds from across Britain *e.g. Black 1986; Philpott 1991; Ramm 1971; Toller 1977*	*Inhumations* Late Roman	All ages Both sexes	Stone sarcophagi; lead-lined coffins	Some containing plaster; many accompanied by high quality grave goods	None reported	Many missing - left in situ; reburied; lead-liners lost/re-used/stolen **Not found/accessed**
Mersea Island, Essex *Discovered 1913* *Residues observed 2013 McKinley 2013, pers. comm., 10-27 March*	*Cremation* Late 1st-mid 2nd	Mature adult Male	Green glass vessel in lead ossuary within tile chamber below barrow	None reported	Observed during analysis of human remains in 2013	Extant; curated by Mersea Museum Trust **SAMPLED 2013**

It is, therefore, clear that the rite of interest, essentially the ‘package’ identified by Ramm (1971), formed part of these imported practices and was substantively different to the ephemeral approach favoured in the PRIA (**4.4**). This is most evident in the material aspects of the treatment of the body as these individuals were placed in stone sarcophagi and/or lead-lined coffins, sometimes with plaster body-casings, accompanying textiles (shrouds and garments) and, on occasions, were located within mausolea. This method of body treatment was designed to endure. Nonetheless, links with earlier practices can be discerned, again in terms of the materials employed. For example, the most elaborate cremation burials in the later PRIA and early Imperial period were marked by deposition in high quality glass urns placed within lead ossuaries in stone containers and/or tiled vaults often at the centre of barrows. Thus, demarcation of difference in response to aspects of the deceased’s identity, in particular their social status, can be seen to extend across the change in the dominant method of disposal within the Roman Empire.

Indeed, despite vast differences in other aspects of mortuary practices, this differentiation in death can be viewed as common to both PRIA and Roman eschatology. A wide variety of methods were employed both before and after the conquest with the element of display a key part of the treatment of the bodies of significant individuals. Conspicuous consumption during the funerary rites of members of the elite and the manipulation of human remains, literally (e.g. retention of the heads) or figuratively (e.g. through the use of *imagines*) in order to maintain a relationship between the living and the dead are attested in the primary sources. Even the concept of using plant products as preservatives, as cited above (**4.2**) was not unknown among the peoples of Gaul. Thus, as “items which were offered on the pyre during cremation [seem subsequently to have been]...placed intact in the grave with the deceased” (Philpott 1991: 224) resinous plant exudates may have been included in inhumation burials. According to classical authors, they certainly played a significant role throughout the rites surrounding earlier cremation burials (**3.2**). In addition, a growing body of evidence from continental Europe supports the contention that they accompanied those inhumed into the tomb (**3.3**).

But did these exotic substances reach the remote province of *Britannia*? A number of researchers have expressed the opinion that this is possible or even probable but proof was not pursued since the general consensus appears to have been that none would survive. Opinions expressed range from statements that “evidence of this act [i.e. the use of unguents] has not, of course, survived in Britain” (Alcock 1980: 62) to laments that “organic [materials] have for the most part disappeared and could, of course, have been valuable” (Esmonde Cleary 1992: 37) and that “[decay] removes...an unknown but probably important component of the original funerary deposits” (Philpott 1991: 1). They also include, however, the insightful comment that there is need for more work in “reconstructing aspects of the burial rite that are usually archaeologically invisible” (Philpott 1991: 5).

This aim of reconstruction is a pressing concern since recent advances in mortuary theory have shown that material evidence is paramount in unlocking the ‘meaning’ encapsulated in the burial record. The potential of invisible chemical evidence is, however, still only poorly understood by many working within both the archaeological and cultural heritage communities (**6.2; 8.2**). It seems likely, therefore, that considerable amounts of information are being lost due, for example, to the discard of deposits suitable for sampling. Before this can be rectified and the finer details of the treatment of the body in death be revealed, the facts regarding the physical and chemical nature of substances of interest and their degradation pathways must be established. Theory is of little value unless we fully understand the materials under scrutiny (Ingold 2007).

Chapter 5

Plant exudates: chemistry and occurrence

"The term resin is not easy to define...for those substances that are commonly termed resins differ a good deal...both in physical properties and in chemical composition".
Vegetable gums and resins, Howes 1949: 85

5.1 Introduction

A large number of trees, shrubs and other plants produce natural exudates, although those species that extrude sticky, often highly scented, secretions in any abundance are rather more restricted (Howes 1949: 3-4, 86-87; Langenheim 2003: 51-105). Since prehistory, many of the latter have been put to a wide variety of anthropogenic uses as adhesives, protective coatings, varnishes and illuminants with the more fragrant varieties used in perfumes, unguents, medicines, embalming and as incense (Colombini and Modugno 2009; Serpico 2000). Although such substances may be categorised in a variety of ways, the simplest approach is a division based on their major chemical components. Under this system, they can be grouped into 'true' resins (e.g. conifer exudates, elemi, mastic) which are predominantly composed of water-insoluble terpenoids, gums (e.g. almond, gum Arabic, tragacanth) which consist mainly of water-soluble polysaccharides and gum-resins (e.g. frankincense, myrrh) which contain variable amounts of both fractions. In addition, low molecular mass (LMM) phenolic compounds occur in many resinous exudates and may, on occasion, comprise their main components (e.g. in benzoin) (Langenheim 2003: 23-50). They are also present, alongside LMM terpenes, in what are termed essential oils which can be extracted from many plants and provide the solvent for higher molecular mass (HMM) triterpenic moieties in balsamic (oleo) resins (e.g. storax, styrax) (Colombini and Modugno 2009; Langenheim 2003: 27).

Two major reviews regarding the sources, occurrence and exploitation of a wide range of these plant products have been undertaken by Howes (1949) and, more recently, by Langenheim (2003). Aspects relating to individual species are detailed in the vast phytochemical literature (see relevant sections below) with additional compositional (e.g. structural characteristics) and mass spectral data provided in many publications dealing with art and archaeological research (e.g. Colombini and Modugno 2009; Mills and White 1999: 95-109; Pollard and Heron 2008; references below). In relation to the current project, this array of botanical sources can be restricted to those native to or otherwise available in antiquity within the Mediterranean region. A summary of those of interest is given in Howes (1950) with a discussion of their chemistry provided by Serpico (1996). For the Roman period, these findings can be supplemented by information from classical sources regarding patterns of use and access via trade networks (e.g. *Anon PME* nd; *Pliny NH 12.24-63* nd; *Theophrastus 9.2-5* nd **3.2**). In addition, previous chemical analyses of archaeological materials have demonstrated the use of specific exudates in Roman mortuary rites (**3.3**).

Despite this wealth of information, numerous difficulties in determining phylogenetic relationships between species abound and considerable gaps in knowledge remain with regards to the complex chemistry of resinous exudates (Langenheim 2003: 23-50; Mills and White 1999: 95-109). Indeed, even the reason and mechanism for the production of natural gums and resins remains somewhat obscure and appears highly variable in terms of output and chemical composition even within a single species (Howes 1949: 3-4; Langenheim 2003: 106-140). Likewise, assigning precise botanical origins to materials described or marketed under vernacular and/or trade names has been shown to be problematic (Howes 1950; Mills and White 1999: 99-109; Regert *et al.* 2008). These challenges are exacerbated in the archaeological record by issues arising from the translation of past names, changes in climate, geographical spread and exudate-production and even the extinction of some species (Gemmill 1966; Loret 1949; Serpico 1996: 22-70). Moreover, the multitude of anthropogenic and environmental impacts on the often poorly-understood degradation pathways of substances employed in the mortuary sphere may obscure links with the original source material (Colombini and Modugno 2009).

These concerns are addressed below in relation to the exudates listed or posited as having played a part in Roman funerary rites based on evidence from the primary sources (**3.2**) and/or their previous identification in mortuary contexts from Europe and North Africa (**3.3**). In order to establish the nature of the materials of interest and the potential for their survival in Britain their chemistry will be considered under the categories: plant gums and essential oils (**5.2**), 'true' resins (diterpenoid and triterpenoid) (**5.3**), gum-resins (**5.4**) and balsamics (**5.5**). The diagnostic characteristics of the principal botanical sources are tabulated below (**Tables 5.1-5.9**). Structural and mass spectral information compiled from the literature and supplemented by the analysis of reference materials (**6.5**) is provided in **Appendix 3.** Key findings are reviewed in **5.6**.

5.2 Plant gums and essential oils

5.2.1 Plant gums

Natural gums are produced by the majority of plant families although species that freely yield significant quantities are more limited (Howes 1949: 3). Those from Europe and Africa that were exploited in antiquity appear predominantly to derive from *Acacia* spp. (gum Arabic), *Astragalus* spp. (tragacanth) and various fruit trees with others possibly traded from the Indian subcontinent (e.g. karaya, guar, ghatti) (Colombini and Modugno 2009; Mills and White 1999: 76-78; Shearer 1989: 348-351). The term gum refers to exudates and seed extracts which display varying degrees of solubility in water ranging from viscous solutions to gelatinous mucilages (Howes 1949: 5-8). The main components of gums are polysaccharides, complex polymeric chains primarily comprised of cyclic aldopentoses and aldohexoses joined by glycoside linkages, often combined with a low level of protein (Bleton *et al.* 1996; Mills and White 1999: 69-73). They are, therefore, readily decomposed via hydrolysis into their component monosaccharides (i.e. sugars such as arabinose, galactose, rhamnose, mannose) and oxidised to produce uronic acids (e.g. galacturonic acid, glucuronic acid) (Colombini and Modugno 2009).

Even in modern samples, these end products are rarely diagnostic and are highly susceptible to leaching (Colombini and Modugno 2009). Thus, although plant gums have been identified as binding media for inks and water-based paints (e.g. Bleton *et al.* 1996; Bonaduce *et al.* 2007; Colombini *et al.* 2002; Lluveras-Tenorio *et al.* 2012), finding evidence in archaeological contexts remains a considerable challenge (Mills and White 1999: 78-80). Only under highly favourable preservation conditions (e.g. arid environments) have traces of gum components been revealed although definitive identification of source has generally proved problematic (Newman and Halpine 2001). Most of these finds relate to the use of gums as paint binders (e.g. Scott *et al.* 2009) although they also seem to have been employed in the construction of cartonnage mummy casings (Wright and Wheals 1987) and to make repairs, for example, by sealing cracks in a sarcophagus (Masschelein-Kleiner *et al.* 1968). In addition, traces of sugars have been observed during the analysis of residues obtained from non-human Egyptian votive mummy bundles (Buckley *et al.* 2004).

Consideration of these factors in relation to the British climate and the lack of references to the use of gums (*per se*) in the Roman mortuary sphere (**3.2.2; Table 3.1**; gum-resins are discussed below) resulted in a decision not to directly investigate sugars as part of this research agenda. An associated project was, however, devised to establish a protocol for use at the University of Bradford in appropriate circumstances (Harrison 2015; **Appendix 7.6**).

5.2.2 Essential oils and phenolic extracts

Similar issues pertain when determining the presence of essential oils such as those obtained from the bark of *Cinnamomum* spp. (cinnamon and cassia), leaves of the genus *Aloe* (aloes) and varieties of valerian (nard or spikenard) or the stigmas of *Crocus sativus* (saffron) mentioned in the primary sources (**3.2.2**). Obtained by various methods (e.g. enfleurage, cold pressing or distillation), such extracts predominantly comprise a broad range of low molecular mass (LMM) moieties (e.g. Ahmed *et al*. 2016; Leela 2008; Serpico 2000; Shelton 1991). These include phenolic acids (e.g. benzoic acid, cinnamic acid) which are often esterified with aryl alcohols or part-bound as glycosides alongside mono- and sesquiterpenes and related compounds (e.g. in cedar oil, see below **5.3.2; Table 5.1**).

Table 5.1. Some of the more characteristic phenolics present in the products of interest (Author, structures from Royal Society of Chemistry (RSC) 2015); TMS = trimethylsilyl derivatives.

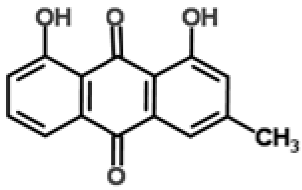
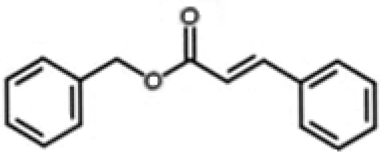
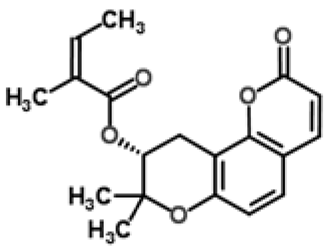
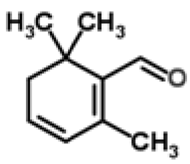
Common name **IUPAC name**	**Chemical structure**	**Formula** **Mass**	**Occurence**
Chrysophanol 1,8-Dihydroxy-3-methyl-9,10-anthraquinone		$C_{15}H_{10}O_4$ 254 TMS 398	*Aloe* spp., cinnamon, cassia extracts
Cinnamaldehyde 3-phenylacrylaldehyde		C_9H_8O 132 TMS NA	*Aloe* spp., cinnamon, cassia extracts
Cinnamic acid 3-phenylacrylic acid		$C_9H_8O_2$ 148 TMS 220	*Aloe* spp., cinnamon, cassia extracts
Cinnamyl acetate 3-phenyl-2-propen-1-yl acetate		$C_{11}H_{12}O_2$ 176 TMS NA	Cinnamon + cassia bark extracts
Benzyl cinnamate benzyl-3-phenylprop-2-enoate		$C_{16}H_{14}O_2$ 238 TMS NA	Cinnamon + cassia bark extracts
Jatamansin 8,8-dimethyl-2-oxo-9,10-dihydropyrano[2,3]chromen-9-yl-2-methylbur-2-enoate		$C_{19}H_{20}O_5$ 328 TMS NA	Valerian extracts e.g. nard
Safranal 2,2,6-trimethyl-4,6-cyclohexadien-1-aldehyde		$C_{10}H_{14}O$ 150 TMS NA	Saffron oil

Table 5.2. Botanical source, geographical spread and chemistry of essential oils employed in the Roman mortuary sphere as recorded in classical sources (**3.2**).

Common name/Taxonomy	Occurrence	Chemistry	References
Aloes Family: Asphodelaceae/Aloaceae Genus: *Aloe* spp.	Southern Africa Madagascar Indian Ocean Islands Arabian Peninsula	Phenolic glycosides e.g. barbaloin and *iso*barbaloin Phenolic compounds e.g. anthraquinone/anthrone, pyrone and chromone derivatives including chrysophanol, chrysophanone and emodin Free and esterified phenolic acids e.g. cinnamic and salicylic acid	Shelton 1991 Vogler and Ernst 1999
Cassia Family: Lauraceae Genus: *Cinnamomum* spp.	India Sri Lanka SE Asia China	Phenolic compounds e.g. anthraquinone/anthrone derivatives including cinnamaldehyde, chrysophanol, chrysophanone and emodin Monoterpenes e.g. cubebene, limonene and terpineol Sesquiterpenes, many with eudesmane skeletons and derivatives Free and esterified phenolic acids e.g. cinnamic acid	Alakshmi *et al.* 2013 Leela 2008
Cinnamon Family: Lauraceae Species: *Cinnamomum verum*	Southern India Sri Lanka	Phenolic compounds dominated by cinnamaldehyde (in bark extract) and eugenol (leaf extract) with cinnamyl acetate and benzyl cinnamate Monoterpenic compounds e.g. borneol, limonene, linalool and α-terpineol Sesquiterpenes and derivatives e.g. β-caryophyllene, caryophyllene oxide and nerolidol Free and esterified phenolic acids e.g. cinnamic acid	Jakhetia *et al.* 2010 Leela 2008
Nard (spikenard) Family: Valerinaceae Species: *Nardostachys jatamansi*	Alpine/subalpine India Pakistan Nepal, Tibet, China	Monoterpenes and derivatives e.g. α- and β-pinene, camphene, limonene and bornyl acetate Sesquiterpenic compounds e.g. aristolone, caryophyllene, maaliene, nardol, patchoulol, valeranone, nardostachone, nardin Coumarins e.g. jatamansin	Disket *et al.* 2012 Purnima *et al.* 2015 Shanbhag *et al.* 1964
Saffron Family: Iridaceae Species: *Crocus sativus*	Eastern Mediterranean Greece + Islands Iran, India, China	Cyclic compounds e.g. safranal (major component in essential oil), α- and β-isophorone, ionones and pyrones (from degradation of carotenoids) Terpenic compounds e.g. linalool Non-volatile compounds dominated by carotenoids e.g. α- and β-carotenes, α-crocin and picocrocin and anthrocyanins [NB: not accessible by GC-MS]	Ahmed *et al.* 2016 Srivastava *et al.* 2010 Tarantilis and Polissiou 1997

Although their solubility in water is low, these molecules are highly volatile and only likely to be retained over archaeological time in exceptional circumstances (Serpico 2000). Many are also non-diagnostic (of common occurrence so they cannot be considered biomarkers for specific exudates) as demonstrated by overlaps in the chemical composition of even the limited number of extracts reportedly employed in Roman funerary contexts (**Chapter 3**; **Table 3.1**) whose main components are listed in **Table 5.2**. In addition, phenolic compounds are released during the natural degradation of high molecular mass (HMM) non-volatile components of plant tissues (e.g. lignin and flavonoids) so, in mortuary contexts, these end products could derive from a number of sources (Pournou 2008; Regert *et al.* 2001).

Likewise, other resinous extracts known in antiquity such as ammoniacum (*Ferula* or *Dorema* spp.), galbanum (*Ferula* spp.) and opopanax (*Opopanax* spp.) could prove difficult to identify with confidence (Hamm *et al.* 2004). In the main, these substances contain low abundance, volatile mono- and sesquiterpenes and associated alcohols of relatively widespread occurrence (Bleton and Tchapla 2009). Generally utilised in medicinal preparations or perfumes, some may also have been employed in the ritual sphere as incense or in scented unguents (Howes 1950; Langenheim 2003: 412-416). Nonetheless, until recently, the only evidence for the use of essential oils has come from Egyptian embalming materials with most of coniferous origin (e.g. Buckley *et al.* 2004; Koller *et al.* 2003; see below **5.3.2**) although the use of galbanum in the treatment of a Ptolemaic period mummy has also been posited (Benson *et al.* 1978). Such finds demonstrate that, when present in considerable abundance and/or stabilised as part of a perfumed mixture, volatile compounds can survive in the archaeological record in certain environmental conditions (e.g. arid or waterlogged; Serpico 1996: 84).

Thus, although unlikely to persist in most British contexts (e.g. in comminuted grave deposits, **6.2**) the possibility remains given suitable circumstances. Indeed, such LMM components have been identified in association with protected Roman period burials in continental Europe. These comprise monoterpenic moieties revealed by pyrolysis (py)-GC-MS in samples from the unusual environment provided by the mass graves in the catacombs of *Santi Marcellino e Pietro*, Rome (Devièse 2008: 121-124). A range of phenolic compounds (e.g. benzoic acid, cinnamic acid and derivatives) have also been observed in the exceptionally well-preserved resinous deposits obtained from sarcophagus burials below St Maximin's, Trier, Germany. In the latter case, these appear to be associated with triterpenic components and so may represent a balsamic resin (Reifarth 2013: 91-93; **5.5**) although an admixture of resinous exudates, oil of cinnamon/cassia or even beeswax cannot, as yet, be precluded. In addition, chrysophanol identified in association with many of silk fabrics from Trier and thought to derive from a dye-stuff (Reifarth 2013: 79-80) could equally represent the impregnation of these textiles with essential oils (e.g. extract of aloes or cassia/cinnamon).

5.3 Resins

5.3.1 Classification

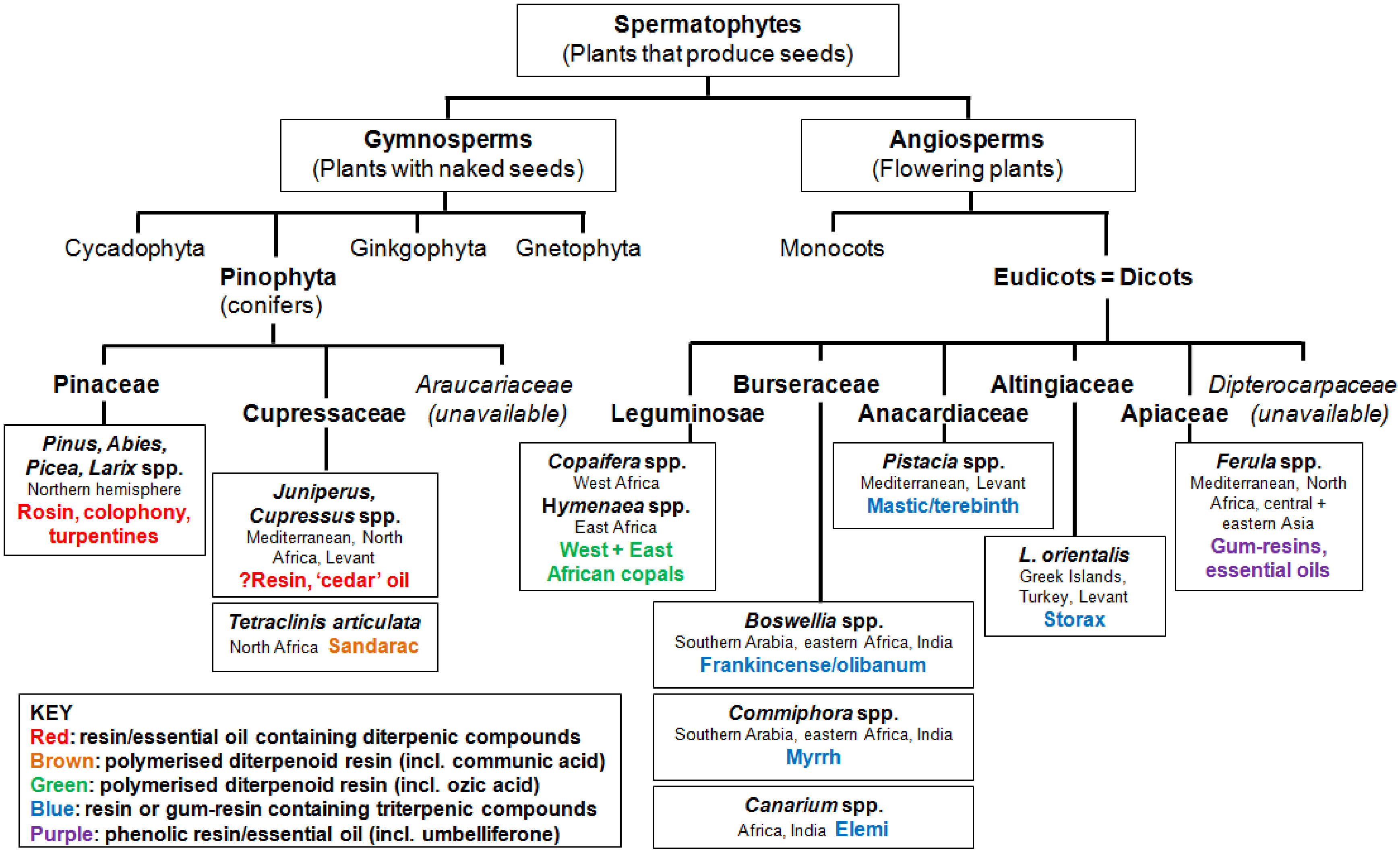
Natural resins are complex materials composed of a range of compounds of varying volatility (Howes 1949: 87-89). The majority of these are terpenic moieties, polycyclic hydrocarbons with oxygen-containing functional groups (terpenoids) and their derivatives, which are classified according to the number of C_5 isoprene ($CH_2{=}C(CH_3)CH{=}CH_2$) units from which they are formed (Pollard and Heron 2008: 241-242; **Table 5.3**). As with the components of polysaccharide gums and essential oils, the LMM weight compounds (mono- and sesquiterpenoids and their derivatives) can rarely act as biomarkers and tend to be lost over archaeological time (Scalarone *et al.* 2003). Moreover, their relative proportions have been shown to be highly variable, even within a single species, as a result of growing conditions, maturation stage and harvesting methods (Alma *et al.* 2004; Assimopoulou and Papageorgiou 2005a, 2005b; Flamini *et al.* 2004).

The terpenoids of diagnostic value are, therefore, primarily the HMM di- and triterpenoids as these are limited in their botanical origins due to the different biosynthetic pathways employed by various plant families (Langenheim 2003: 24-30). These two categories of terpenoids are rarely produced by the same species (Pollard *et al.* 2007: 154) and permit the characterisation of different taxa, usually to the level of genus, since commonality in components and the homogenising impact of degradation pathways means that more precise botanical identification remains elusive even in modern materials (Langenheim 2003: 34-40; Mills and White 1977). Fortunately, they also comprise the more degradation resistant, water-insoluble fraction of 'true' resins, certain gum-resins and balsams and so evidence of their presence may survive in the archaeological record (Colombini and Modugno 2009).

Table 5.3. Classification of terpenoids by carbon number, characteristics and occurrence in resinous exudates (after Langenheim 2003: 34-40; Mills and White 1999: 95-115).

Terpenoid group	Carbon number	Characteristics	Occurrence in exudates
Monoterpenoids	10	Highly volatile Distinctive odour Of widespread occurrence Presence/absence and percentage abundance naturally variable	Present in both conifer and angiosperm exudates More prevalent in coniferous exudates
Sesquiterpenoids	15	Volatile Distinctive odour Of widespread occurrence Presence/absence and percentage abundance naturally variable	Present in both conifer and angiosperm exudates More prevalent in angiosperm exudates
Diterpenoids	20	Relatively non-volatile Different skeletal series (e.g. abietane, pimarane, labdane) diagnostic of different families	Characteristic of conifers: Pinaceae, Cupressaceae Also present in Leguminosae (e.g. *Hymenaea* spp.) and Cistaceae exudates
Sesterterpenoids	25	Very rare	No known significance
Triterpenoids	30	Relatively non-volatile Different skeletal series (e.g. dammarane, tirucallane, oleanane, ursane, lupane) diagnostic of different families	Characteristic of angiosperms: Burseraceae, Anacardiaceae, and some balsamic (e.g. *Liquidambar orientalis*) resins. Also present in some gum-resins (e.g. *Boswellia* spp.)
Polyisoprenoids	$(C_5)_n$ polymers	Polymerised portion Non-volatile [not accessible by GC-MS]	Occur in most resins Age/environment related

Figure 5.1. Botanical taxonomy, geographical occurrence and basic chemistry of resin-producing plants available within the Roman Empire and mentioned in the text (Figure modified from Regert *et al.* 2008: Figure 1, 671).



Although the tissues of many plant species contain a range of terpenic compounds, the main resin-producing families are the conifers (Pinaceae, Cupressaceae, Araucariaceae) and certain angiosperms (mainly the Anacardiaceae, Burseraceae, Dipterocarpaceae, Leguminosae and Styraceae) (Langenheim 2003: 35-40; **Figure 5.1**). Research has shown that resins from conifer species are characterised by a large volatile fraction (mono- and sesquiterpenes) combined with non-volatile diterpenoid acids with three main skeletal types (abietane, pimarane and/or labdane), depending on family (Mills and White 1977). In contrast, resins produced by angiosperms generally have volatile fractions dominated by sesquiterpenes and non-volatile fractions comprised predominantly of triterpenoids. The exceptions are the Leguminosae (e.g. *Hymenea* spp.) and Cistaceae (*Cistus* spp.) which contain labdane-type diterpenic acids (Langenheim 2003: 37-40). Quantitative and even qualitative variability, including of the HMM terpenic components, within each genus or species can, however, be considerable often as a result of environmental factors (Mills and White 1999: 95-109).

This complexity is further complicated in archaeological materials by natural aging processes such as oxidation and anthropogenic activities such as heating or mixing which can alter the relative abundances and chemical structures of key compounds (Serpico 2000). Thus, degradation pathways, which will have modified the initial 'fingerprint' of the resin, have to be considered (Charrié-Duhaut *et al.* 2009) and suites of biomarkers used to securely characterise ancient resinous exudates (Evershed 2008a). These issues are discussed below in relation to diterpenoid (**5.3.2**) and triterpenoid (**5.3.3**) resins with reference to their previous identification in archaeological contexts (N.B. common names for compounds are used in the text for brevity. IUPAC nomenclature is given for key biomarkers in relevant Figure and Table captions and in **Appendix 3**).

5.3.2 Diterpenoid-containing resins

A number of plant families produce exudates containing diterpenic compounds with those of interest comprising members of the Pinaceae, Cupressaceae, Leguminosae and Cistaceae (Mills and White 1999: 99-105;

Serpico 1996: 25-40, 55-58; **Table 5.4**). Of these, the most widely utilised resin-producing species belong to the taxonomic division, the Coniferae, predominantly those in the Pinaceae (e.g. pine, cedar) and, to a lesser extent, the Cupressaceae (e.g. juniper, cypress) families (Mills and White 1977). The natural exudates of these gymnosperms (seed cone bearing trees and shrubs) have been employed as sealants, adhesives, illuminants and in wine with their resinous softwoods heated to provide tars and pitches and often referred to as 'naval stores' due to their waterproofing properties (Howes 1949: 104-110; Langenheim 2003: 306-331). As a consequence of this extensive exploitation, the resin chemistry of the conifers has been relatively well characterised which has facilitated their identification in the archaeological record (Colombini and Modugno 2009). Thus, evidence for Pinaceae products, in particular, has been obtained from a range of contexts (e.g. Colombini *et al.* 2003, 2009; Connan and Nissenbaum 2003; Evershed *et al.* 1985; Hjulström *et al.* 2006; Romanus *et al.* 2009).

In the Roman period, the resinous woods and extracts of the Pinaceae appear to have been used, largely, in shipbuilding and to line *amphorae* and other containers (e.g. Colombini *et al.* 2005; Cramp *et al.* 2011; Evershed 1993; Heron and Pollard 1988). These applications are described by Pliny (*NH 16.16-22* nd) who also notes their ritual connotations, with the pitch tree (*Picea* spp.?) perceived as "*a funeral tree...placed at the doors of houses as a token of bereavement and grown on graves*" and the more fluid and abundant exudate from the torch tree (*Pinus* spp.?), used "*for torches at religious ceremonies*" and "*in Egypt, for embalming the dead*". The latter statement is corroborated by chemical evidence obtained from the analysis of mummy balms (e.g. Colombini *et al.* 2000; Jones *et al.* 2014; Koller *et al.* 1998; Łucejko *et al.* 2012; Maurer *et al.* 2002; Proefke and Rinehart 1992) with indications for the increased use of Pinaceae resins in the Ptolemaic and Roman periods (Buckley and Evershed 2001; Corcoran and Svoboda 2010). Indeed, the pine appears to have held a special significance in Roman eschatology with pinecones, as symbols of immortality or mourning, found as finials and carvings on funerary monuments (Alcock 1980; Mackinder 2000:

14-16). The presence of biomarkers denoting the use of diterpenoid resins in Roman mortuary contexts in continental Europe adds support to this inference with some individuals from Trier also found to have been interred with *Abies* spp. (fir) wood shavings (Ascenzi *et al.* 1996; Devièse *et al.* 2010; Papageorgopoulou *et al.* 2009; Reifarth 2013: 29-30; **3.3.4**).

Distributed across the northern hemisphere, all members of the Pinaceae yield exudates characterised by a non-volatile fraction consisting of diterpenic compounds (Mills and White 1999: 99). Of these, pines (*Pinus* spp.) are both the largest group and the most abundant resin producers (often separated by distillation into oil of turpentine, the fluid fraction, and rosin/colophony, the solid fraction) (Howes 1949: 54-59, 104-110; Langenheim 2003: 54-64). The resinous woods and possibly the essential oils of *Cedrus* spp. ('true' cedars) have also been employed although there is little evidence for the extensive utilisation of *Abies* spp. (firs), *Picea* spp. (spruces), and *Larix* spp. (larches) products in antiquity (Howes 1949: 154-165; Langenheim 2003: 321-331, 343). In terms of their chemistry, the exudates secreted by these genera are very similar with the diterpenoids principally consisting of abietane and pimarane skeletal forms (Mills and White 1999: 99; Otto *et al.* 1997). *Abies* spp. and *Larix* spp. have also been found to incorporate neutral labdanes (e.g. *cis*-abienol, epimanool, larixol, larixyl acetate) which can help distinguish them (Baumer *et al.* 2009; Mills and White 1999: 100-102; Scalarone *et al.* 2002) while an abundance of pimaric and abietic acid isomers is more characteristic of *Pinus* spp. exudates (Mills and White 1977; Colombini *et al.* 2000; **Figure 5.2**).

Although the resin acids (e.g. pimaric, sandaracopimaric, *iso*pimaric and abietic acid) dominate in fresh resins, they only tend to survive over archaeological time in relatively well preserved materials (Serpico 1996: 79-80). Identification is, therefore, often based on the presence of their more persistent intermediate (e.g. dehydroabietic, didehydroabietic and 7-oxo-dehydroabietic acids) and/or final stage degradation products (e.g. retene and methyl dehydroabietate) (Colombini and Modugno 2009). These changes largely result from oxidation processes, with naturally aged

Pinaceae resins characterised by increased levels of functionalised (i.e. still retaining their acid groups) and defunctionalised abietane-skeleton compounds (e.g. norabietadienes, norabietatetraenes) since pimaranes, although their isomeric acids are initially more stable, appear more readily lost over longer time periods (Mills and White 1977; Pastorova *et al.* 1997). As these pathways result in increased homogeneity, taxonomic classification of conifer resins is best restricted to the level of family, i.e. Pinaceae.

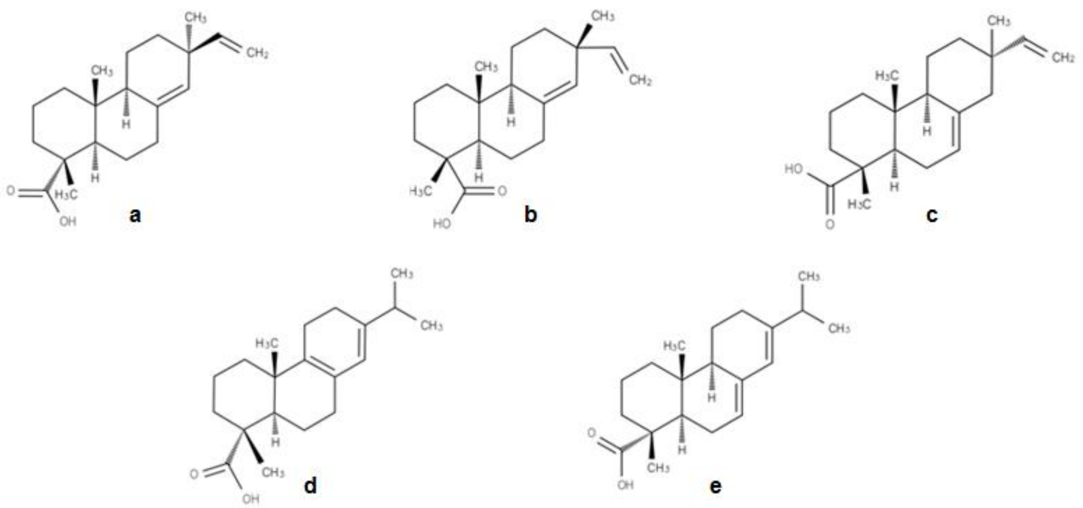


Figure 5.2. Diterpenic compounds characteristic of conifer resins: a. pimaric acid (13β-pimara-8,15-dien-18-oic acid); b. sandaracopimaric acid (13α-pimara-8,15-dien-18-oic acid) often in considerable abundance in Cupressaceae resins; c. *iso*pimaric acid (pimara-7,15-dien-18-oic acid); d. palustric acid (abieta-8,13-dien-18-oic acid) only present in Pinaceae resins; e. abietic acid (abieta-7,13-dien-18-oic acid) only present in Pinaceae resins (Author, structures from European Molecular Biology Laboratory-European Bioinformatics Institute (EMBL-EBI) 2016 and National Institute of Standards and Technology (NIST) 2016).

The presence/absence and relative proportions of some of these compounds have also been used to distinguish between natural environmental degradation and anthropogenic pre-treatment processes such as heating to produce tars and pitches (Connan and Nissenbaum 2003). Experimental studies have shown that thermal degradation leads to dehydrogenation, decarboxylation and a higher degree of aromatisation of the cyclic compounds present (Egenberg *et al.* 2002). As with environmental degradation, a mixture of DHA acid and various neutral compounds (intermediates such as tetrahydroretene, norabietatrienes and norabietatetraenes) is produced although, as the degree of alteration is temperature dependent, presence/absence and relative abundance tends to be highly variable (Beck *et al.* 1994). Nonetheless, in Pinaceae resin tars retene (RET) appears to be the final stable product (Robinson *et al.* 1987) whereas significant levels of methyl dehydroabietate (MDHA) may indicate the pyrolysis of resinous Pinaceae woods (Colombini *et al.* 2003). The latter compound results from a reaction between methanol, released by the heating of the wood, and the diterpenoid acids present but it may also be formed through reactions within in the burial environment (Colombini and Modugno 2009). In addition, sample preparation procedures (e.g. solvent extraction using DCM:methanol with warming on a hot-plate or derivatisation using methylation) can increase the amount of MDHA making it an unreliable indicator. Caution is, therefore, advised when interpreting such data especially in highly degraded materials. In addition, as the Pinaceae are of extensive modern occurrence across Europe, including Britain, the possibility of environmental contamination must be addressed since traces of these persistent end products (e.g. DHA, MDHA, RET) are often found in soils and sediments (Hjulström *et al.* 2006; **4.2**).

Essential oils obtained from Pinaceae resins and resinous woods also have a long history of anthropogenic exploitation (Langenheim 2003: 306-323). These liquid extracts (often called turpentine) contain a large volatile fraction dominated by monoterpenes (e.g. α- and β-pinene, limonene, β-phellandrene), with a lower abundance of sesquiterpenes (e.g. cadelene, caryophyllene, cuparene, longifolene) and phenolic components (e.g. thymol, guaiacol, benzocycloheptadiene derivatives). Traces of diterpenoids and their derivatives may also be present (Mills and White 1999: 95-97; Serpico 2000). Referred to as ‘cedar oil’ by classical authors (e.g. Herodotus, Pliny, Diodorus Siculus) and employed as a purgative during Ancient Egyptian embalming processes (Baumann 1960), compounds indicative of the use of such conifer extracts have been observed in samples from human and votive (non-human) mummies (e.g. Buckley *et al.* 2004; Corcoran and Svoboda 2010; Koller *et al.* 2003, 2005; Łucejko *et al.* 2012), canopic jars (Charrié-Duhaut *et al.* 2007) and in varnishes applied to coffins and *shabti* figures (Bianucci *et al.* 2015; Serpico and White 2001).

Table 5.4. Botanical source, geographical spread and chemistry of diterpenoid-containing exudates available and/or attested within the Roman Empire.

Product(s)/taxonomy	**Occurrence**	**Chemistry**	**References**
Turpentine (fluid exudate) **Colophony/rosin** (solid) **Tar/pitch** (heated product) Family: Pinaceae Genus: *Pinus* spp. (pines) *Abies* spp. (firs) *Picea* spp. (spruces) *Larix* spp. (larches)	Northern hemisphere Europe, Mediterranean, Asia	Monoterpenic compounds: e.g. α- and β-pinene, limonene, β-phellandrene, himachalenes (*Cedrus* spp.), camphor, linalool Sesquiterpenic compounds: e.g. junipene, cadalene, cadinatriene, cuparene Diterpenic compounds: pimaranes and abietanes (in all genera) e.g. pimaric (not *Abies* spp.), sandaracopimaric, isopimaric and abietic acid with neutral labdanes in *Larix* spp. e.g. epimanool, larixol, larixyl acetate	Baumer *et al.* 2009 Koller *et al.* 2005 Mills and White 1999: 95-102 Otto *et al.* 1997 Scalarone *et al.* 2002
Cedar(wood) oil/resin Family: Cupressaceae Genus: *Juniperus* spp. (junipers) *Cupressus* spp. (cypresses) **Sandarac** Family: Cupressaceae Species: *Tetraclinis articulata*	Worldwide Europe, Africa, Asia Worldwide Mediterranean, North Africa	Phenolic compounds: e.g. totarol and ferruginol Sesquiterpenic compounds: α- and β-cedrene, cuparene, cadinene, thujopsene, cedrol Diterpenoids and derivatives: pimaradienes e.g. sandaracopimaric acid; labdanes e.g. (poly)communic acid Diterpenoids and derivatives: e.g. sandaracopimaric acid, sandaracopimarinol, (poly)communic acid	Howes 1949: 136-137 Mills and White 1977 Otto and Wilde 2001
Copals/copaibas (African) Family: Leguminosae Genus: *Hymenaea* spp. *Copaifera* spp.	Tropics Tropical Africa	Sesquiterpenic compounds e.g. β-caryophyllene, α- and β-humulene, α- and β-copaene and δ-cadinene Diterpenic compounds: labdadienes (polymerised), kauranes and clerodanes e.g. ozic, zanzibaric, pinifolic and copalic acid	Colombini and Modugno 2009: 19 Langenheim 1996 Leandro *et al.* 2012
Labdanum Family: Cistaceae Genus: *Cistus* spp. (rock roses)	Mediterranean Mediterranean	Phenolic compounds: e.g. dihydrocinnamic acid Sesquiterpenic compounds: e.g. aromadendr-1-ene, ledene Diterpenic compounds: labdanes and clerodanes e.g. laurifolic, labdanolic and cistenolic acid	Hamm *et al.* 2004 Papaefthimiou *et al.* 2014

There has, however, been considerable debate concerning the precise botanical source of the ‘cedar oil’ employed in antiquity with extracts from other members of the Pinaceae (e.g. pine, fir, spruce) proposed alongside those obtained from *Cedrus* spp. (Koller *et al.* 2005). Indeed, due to the low oil yield of many ‘true’ cedars, most modern cedar/cedar wood extracts are derived from members of the Cupressaceae (e.g. junipers, Langenheim 2003: 329-331). It has, therefore, been suggested that this might also have been the case in the past and/or that ‘cedar oil’ was simply a generic term for any coniferous essential oil (Baumann 1960; Lucas and Harris 1962: 306, 320).

With regards to the Cupressaceae, those native to the Mediterranean and northern Africa comprise a number of *Juniperus* spp. alongside *Cupressus semprevirens* and *Tetraclinis articulata*, the source of sandarac (Langenheim 2003: 382-385; Serpico 1996: 35-40). Exudates produced by this family can be distinguished from those of the Pinaceae due to a high abundance of sandaracopimaric acid and related pimaradiene acids and/or labdane-skeleton compounds such as communic acid (Howes 1949: 136-137; Mills and White 1977; **Table 5.4**). The latter, however, is readily polymerised to polycommunic acid which is the major component in most Cupressaceae resins (c. 70% in sandarac) (Mills and White 1999: 102-103). This high polymer content limits the effectiveness of GC-MS and, even though breakdown has been noted in aged in materials, similarities in diterpenoid content (including with larch) restricts lower level botanical identification (Baumer *et al.* 2009). Recourse can be made to characteristic phenolic compounds (e.g. totarol, ferruginol), if present, although these also occur in *Cedrus atlantica* (Otto and Wilde 2001; Serpico 1996: 81-82). Thus, indications for the past use of Cupressaceae resins are infrequent (Baeten *et al.* 2014; Deviése *et al.* 2010; Reifarth 2013: 110) and it seems that it may have been their aromatic woods that were more commonly employed in ritual contexts (e.g. wadding of mummies, manufacture of coffins and roofing of temples) (Baumann 1960; Howes 1949: 137; Gale *et al.* 2000).

Diterpenic compounds are also present in exudates obtained from members of the Leguminosae (e.g. *Hymenaea* spp., African copals; *Copaifera* spp., African copaibas) and *Cistus* spp. (labdanum) (Mills and White 1999: 103-105; Langenheim 2003: 435-437). The copals/copaibas contain a range of sesquiterpenes (e.g. β-caryophyllene, α- and β-humulene, α- and β-copaene, δ-cadinene) and distinctive diterpenoids with labdane, kaurane and clerodane-skeletal structures (e.g. ozic, *iso*ozic, zanzibaric, pinifolic, hardwickiic and copalic acids) (Langenheim 1996; Leandro *et al.* 2012). Identification in aged materials has, however, been hampered by natural variability, the evaporation (sesquiterpenes), ready polymerisation (diterpenoids) and the range of isomers and enantiomers (e.g. of communic acid) in these exudates (Mills and White 1999: 103-105; Serpico 2000: 454; Scalarone *et al.* 2003; van der Werf 2000). In addition, taxonomic relationships and links between common names and botanical source are particularly obscure with modern exudates often, erroneously, referred to as amber (Langenheim 2003: 144-147). Nonetheless, *Hymenaea* spp. resins have recently been identified from medieval contexts in Tanzania and Yemen (Crowther *et al.* 2015; Regert *et al.* 2008).

Likewise, resinous exudates containing compounds of relatively rare occurrence can be obtained from the leaves and twigs of rock roses (members of the Cistaceae which in countries and islands of the Mediterranean include *Cistus creticus, C. laurifolius* and *C. ladanifer*) (Howes 1950; Serpico 1996: 55-58). The majority of these potential biomarkers are, however, either highly volatile phenolics (e.g. dihydrocinnamic acid) or terpenes (e.g. aromadendr-1-ene, ledene) and/or are present in relatively low abundance (e.g. labdane and clerodane diterpenoids such as laurifolic acid, labdanolic acid and cistenolic acid) (Hamm *et al.* 2004; Papaefthimiou *et al.* 2014). Known as labdanum, these exudates have yet to be securely identified in the archaeological record (Lucas and Harris 1962: 94; Serpico 2000; Tchapla *et al.* 2004).

5.3.3 Triterpenoid-containing resins

'True' resins produced by angiosperms (gum-resins and balsams are discussed below) other than those obtained from the Leguminosae and Cistaceae (see above, **5.3.2**) contain a range of tetracyclic (e.g. dammarane, euphane or tirucallane skeletons) or pentacyclic (e.g. ursane, oleanane or lupane skeletons) triterpenic compounds (**Figure 5.3**). Those native to the Mediterranean region or available through trade seem to have been restricted to members of the Anacardiaceae (*Pistacia* spp.) and Burseraceae (*Canarium* spp.) (Colombini and Modugno 2009; Langenheim 2003: 38-39; **Table 5.5**). Of these, *Pistacia* spp. resins (mastic/terebinth) appear to have been of considerable significance in antiquity (Mills and White 1977; Serpico 1996). A key ingredient of the unguent and embalming mixture known as *kyphi*, the highly prized exudates of the genus have also been used as incense, in perfumes and in medicinal preparations (Baumann 1960; Howes 1949: 138-139; Langenheim 2003: 388-390). Indeed, sources from the 1st century AD refer to terebinth resin as the finest due to its scent, astringency and colour, with mastic a close second (*Pliny NH 14.25:122* nd; *Theophrastus 9.1:6* nd).

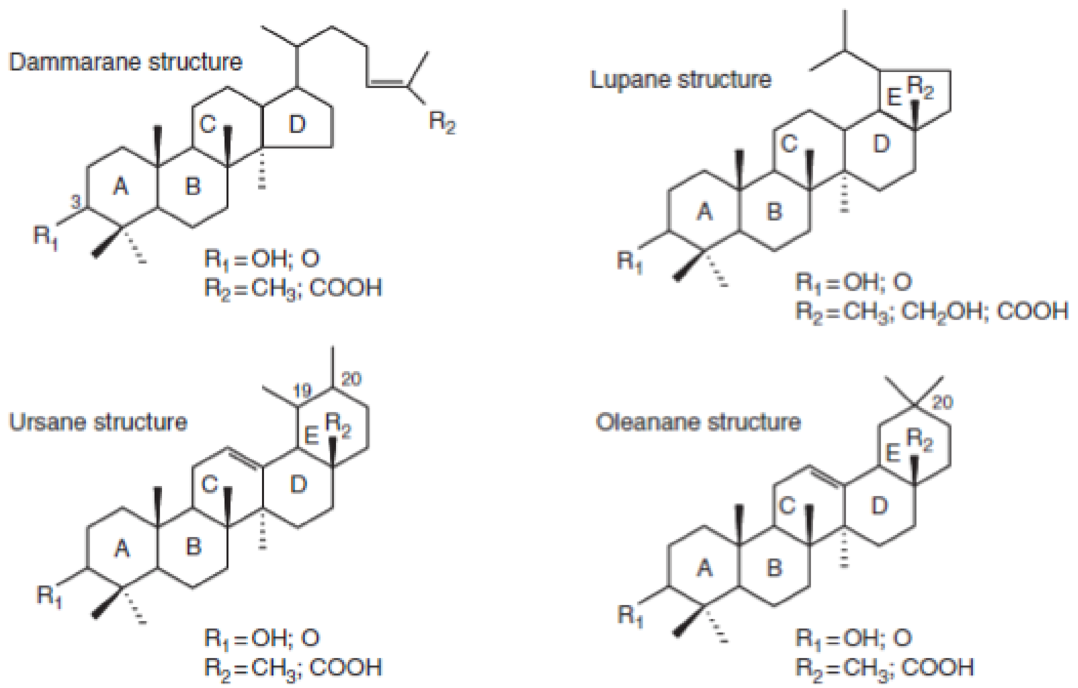


Figure 5.3. General skeletal structures of the main triterpenic families present in angiosperm resins (Figure from Colombini and Modugno 2009: Figure 1.2, 15).

This literary evidence is now supported by the molecular characterisation of *Pistacia* spp. exudates in a range of, predominantly, Egyptian ritual contexts: in varnishes on funerary objects (Serpico and White 2001), as deposits on incense bowls and *amphorae* from New Kingdom temple sites such as Amarna (Serpico and White 2000; Stern *et al.* 2003, 2008a), in embalming materials from Middle Kingdom to Roman period human and non-human mummies (e.g. Buckley and Evershed 2001; Buckley *et al.* 2004; Colombini *et al.* 2000; Ménager *et al.* 2014; Vieillescazes and Coen 1993), mingled with Pinaceae resin and a plant oil in an Etruscan period ointment found in a tomb in Italy (in an *unguentarium*, Colombini *et al.* 2009) and as substantial residues in Canaanite *amphorae* from the Late Bronze Age shipwreck off Uluburun, Turkey (Mills and White 1989; Stern *et al.* 2008b). Moreover, inscriptions on the ceramics from Amarna confirm that the highly prized resin, *snṯr*, referred to in Ancient Egyptian texts was (as previously proposed by Loret 1949), a *Pistacia* spp. rather than a *Boswellia* spp. (frankincense) exudate (Serpico and White 2000). The continued use of mastic/terebinth in mortuary contexts during the Roman period has also recently been demonstrated in finds from Italy and Gaul (Bruni and Guglielmi 2005, 2014; Devièse 2008: 63-65; Reifarth 2013: 108-110; **3.3; Table 3.4**).

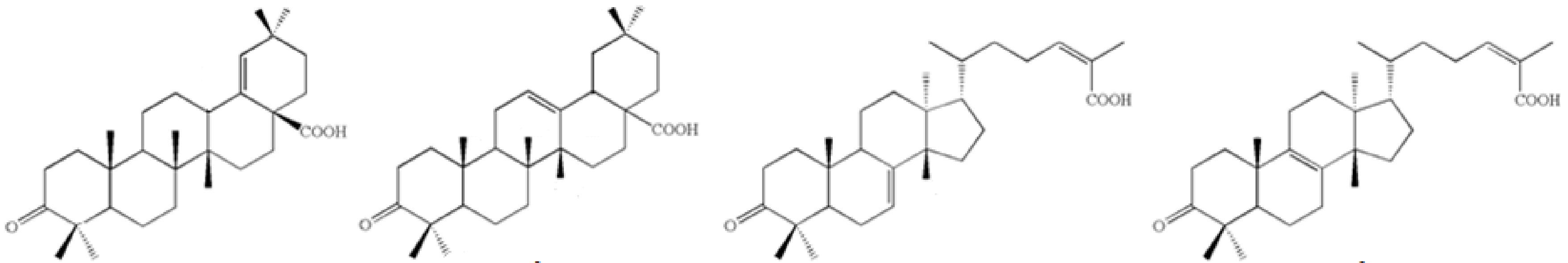
Native to the Mediterranean and extending into the Caucasus and the Levant, around a dozen species of *Pistacia* have been described in the botanical literature (Zohary 1952). The best attested are: *P. vera* and *P. khinjuk* which produce large edible nuts, *P. terebinthus* and its closely related forms *P. atlantica* and *P. palaestina* and the genetically distinctive *P. lentiscus* and its varieties (Parfitt and Badenes 1997). In terms of resin production, *P. lentiscus* var. *Chia*, which grows in a restricted area of the island of Chios is the only species currently exploited commercially (Browicz 1987). Known as mastic, this transparent, yellow exudate, which initially extrudes as a viscous liquid, hardens to brittle ‘tears’ on exposure to air (Langenheim 2003: 385-387).

In antiquity, Chios is also recorded as being the most abundant source of the “*most highly praised...white mastic*” (*Pliny NH 12.36:72* nd) although an even more desirable exudate was obtained from the ‘terebinth’ or ‘turpentine tree’ (*Pliny NH 14.25, 16.22* nd). Described as being “*the best, for it sets firm, is the most fragrant, and has the most delicate smell; but the yield is not abundant*” (*Theophrastus 9.2:2* nd), the latter seems only to have been extracted in viable quantities from a species which grew as a large tree near Damascus, Syria (*Pliny NH 13.12* nd). As two other *Pistacia* species, *P. terebinthus* and *P. atlantica*, are known to produce resins it has been suggested that one of these was the ‘turpentine tree’ (van den Berg 1998; Mills and White 1999: 107-108). Definitive botanical identification has proved difficult, however (Parfitt and Badenes 1997), and the debate has spilled over into the archaeological field (Assimopoulou and Papageorgiou 2005b; Hairfield and Hairfield 1990; Mills and White 1989).

The chemistry of *Pistacia* spp. resins is equally complex and has been subject to a number of detailed molecular studies (e.g. Assimopoulou and Papageorgiou 2005a, 2005b; Langenheim 2003: 387; references therein). This research has demonstrated that fresh exudates contain both mono- (e.g. α- and β-pinene, camphene, limonene, linalool) and sesquiterpenic compounds (e.g. β-caryophyllene) of common occurrence, some polymerised components and a highly characteristic suite of biomarkers (Bleton and Tchapla 2009; Serpico 2000; van den Berg 1998). The latter comprise triterpenoids with, predominantly, oleanane and tirucallane skeletons including moronic, oleanonic, masticadienonic and *iso*masticadienonic acid accompanied by less diagnostic dammaranes (Barton and Seoane 1956; Caputo *et al.* 1978; Marner *et al.* 1991; Papageorgiou *et al. 1*997; **Figure 5.4**). Identification of resins from the genus *Pistacia* can, therefore, be undertaken with some assurance (Modugno and Ribechini 2009). Nonetheless, even in modern samples, it is not yet possible to definitively distinguish between *Pistacia* species although certain low abundance derivatives (e.g. 3,4-seco-28-norolean-12(18)-en-3-oic and olean-18-enolic aldehyde) have not as yet been reported in *P. lentiscus* and so may indicate a resin from *P. terebinthus* (or a closely related species) (Assimopoulou and Papageorgiou 2005a, 2005b).

Table 5.5. Botanical source, geographical spread and chemistry of triterpenoid-containing resins available and/or attested within the Roman Empire.

Product(s)/taxonomy	**Occurrence**	**Chemistry – phenolics and terpenoids**	**References**
Mastic/terebinth resin Family: Anacardiaceae Genus: *Pistacia* spp.	 Africa, Asia, the Americas with extension into Europe Temperate Europe, the Mediterranean and Levant	Monoterpenic compounds: e.g. α- and β-pinene, camphene, limonene Sesquiterpenic compounds: e.g. β-caryophyllene Triterpenic compounds: oleananes e.g. moronic acid, oleanonic acid and their derivatives; lupanes e.g. lupan-3-one; tirucallanes/euphanes e.g. tirucallol, masticadienonic and *iso*masticadienonic acid and their derivatives; ocotillones (oxidised dammaranes) (**Figures 5.3** and **5.4**)	Assimopoulou and Papageorgiou 2005a, 2005b Barton and Seoane 1956 Bleton and Tchapla 2009 Caputo *et al.* 1978 Marner *et al.* 1991
Elemi Family: Burseraceae Genus: *Canarium* spp. Species: *C. schweinfurthii*	 Africa, Asia, the Americas Tropical Africa and Asia Tropical Africa	Phenolic compounds: e.g. elemicin Monoterpenic compounds: e.g. limonene Sesquiterpenic compounds: e.g. α-elemol and β-eudesmol Triterpenic compounds: principally α- and β-amyrin (triterpenols with oleanane and ursane skeletons, respectively) and their derivatives (**Figures 5.3** and **5.5**)	Bandaranayake 1980 Cartoni *et al.* 2004 Mills and White 1999: 108



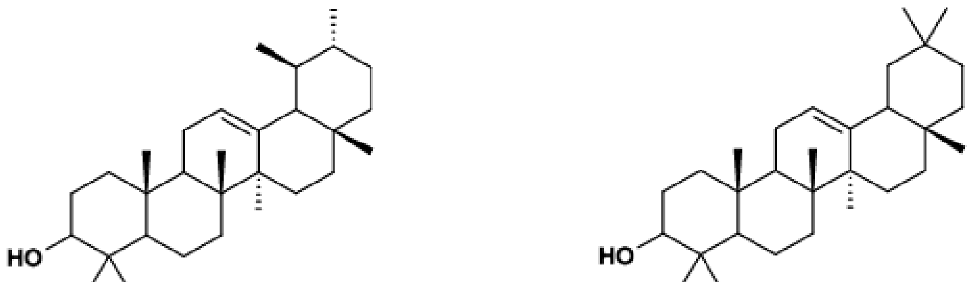
a b c d

Figure 5.4. Skeletal structures of triterpenoid resin acids diagnostic of *Pistacia* spp. resins: a. moronic acid (3-oxoolean-18-en-28-oic acid); b. oleanonic acid (3-oxoolean-12-en-28-oic acid); c. masticadienonic acid (3-oxotirucalla-7,24-dien-26-oic acid); d. *iso*masticadienonic acid (3-oxotirucalla-8,24-dien-26-oic acid) (Author, structures from EMBL-EBI 2016).

The natural degradation pathways of *Pistacia* spp. resins and the potential impact of anthropogenic modification (e.g. heating) on their chemistry have also been evaluated (Modugno and Ribechini 2009). Studies have indicated that the relative proportions of the resin acids (e.g. moronic and oleanonic acid, with an increased abundance of the former indicative of more extensive degradation) may correspond with level of preservation and that the presence/absence of other compounds can aid in determining pre-deposition processes (Mills and White 1999: 108; Serpico and White 2000). Thus, the survival of low abundance or more readily degraded compounds (e.g. tirucallol), generally absent from archaeological materials, may provide an indication that the resin had been deposited in its natural state (i.e. unheated) although inherent variability remains an issue (Assimopoulou and Papageorgiou 2005a). In contrast, an increase in defunctionalised and oxidised derivatives, such as ocotillone-type molecules (oxidised dammaranes with a base peak of *m/z* 143), are often prevalent in naturally aged mastic/terebinth resins (van der Doelen *et al.* 1998).

Likewise, it has been suggested that an abundance of certain degradation products, 28-norolean-17-en-3-one in particular, may indicate that the resin had been heated (Colombini *et al.* 2000; Serpico and White 2000). Unfortunately, this does not appear to be a simple correlation and it has even been argued that 28-noroleanenes are formed during biosynthesis (Assimopoulou and Papageorgiou 2005a). A series of unidentified compounds with a characteristic fragment at *m/z* 453 (in methylated samples) may, therefore, provide a more reliable marker of extensive heating (Stern *et al.* 2003). More work still needs to be done to elucidate this question, however, as gentle warming to increase fluidity/produce a viscous liquid which could be dripped onto the body or pasted onto the textile wrappings may not, as yet, be chemically detectable. Experimental studies and the application of combined approaches (GC-MS in conjunction with FTIR and NMR) may, in future, assist in unravelling the complexities of these degradation pathways (Modugno and Ribechini 2009). Nevertheless, *Pistacia* spp. resins remain one of the most readily characterised exudates in the archaeological record (Stern *et al.* 2003).

The same cannot be said of the resin produced by *Canarium* spp. (Colombini and Modugno 2009). Native to tropical Africa, this exudate obtained from *C. schweinfurthii* is commonly known as elemi, a term also used in relation to members of the Burseraceae (e.g. *Amyris* spp., *Bursera* spp., *Protium* spp.) from the Americas (Howes 1949: 141-144; Langenheim 2003: 356-357). To add to this confusion, the name 'African elemi' has even been applied to the gum-resin of *Boswellia frereana* (Tucker 1986; see below, **5.4**). With respect to *Canarium* spp. elemi, the chemical composition of this powerfully fragranced, malleable, yellow, resin is dominated by two pentacyclic alcohols, α- and β-amyrin (with ursane and oleanane skeletons, respectively; **Figure 5.5**). These triterpenols are dispersed in an abundance of volatile components (e.g. phenolics such as elemicin and mono- and sesquiterpenic compounds including limonene, α-elemol and β-eudesmol) (Bandaranayake 1980; Cartoni *et al.* 2004; Mills and White 1999: 108). A novel class of compounds, for which the name 'canarane' has been proposed, has also recently been identified (Kamdem *et al.* 2011) although these have yet to be characterised by GC-MS.



a b

Figure 5.5. Skeletal structures of triterpenic alcohols dominant in *Canarium* spp. resins (elemi): a. α-amyrin (3β-urs-12-en-3-ol); b. β-amyrin (3β-olean-12-en-3-ol) (Author, structures from EMBL-EBI 2016).

The loss over time of the LMM moieties together with the ubiquity of the triterpenols and their derivatives has made elemi difficult to identify in aged materials and so it is unsurprising that archaeological evidence is scarce (Colombini and Modugno 2009; de la Cruz-Cañizares *et al.* 2005; Mills and White 1977). Of particular interest in terms of this study, however, has been the discovery of amorphous residues adhering to textile fragments, probably shrouds, recovered from Nabatean rock-cut monumental tombs (dated c. 1st century AD) in ancient Hegra (Madâ'in Sâlih, Saudi Arabia). Detailed analysis of a number of samples has indicated that a Burseraceae resin, most closely resembling a *Canarium* spp. exudate, was applied, presumably as part of mortuary rites associated with the treatment of the body (Mathe *et al.* 2009).

5.4 Gum-resins

Gum-resins (or oleo-gum-resins) as their name implies, contain variable percentages of a polysaccharide (gum) fraction and a terpenic (resin) fraction (Langenheim 2003: 362-374). Those available within the Roman Empire consisted of frankincense and myrrh, two of the best known plant exudates in human history regardless of period, place or religious affiliation (Groom 1981; **Table 5.6**). Both are obtained from members of the Burseraceae whose geographical spread, in the 'Old World', extends from the Levant to Arabia, Africa and India (Mills and White 1999: 108-109). Thus, frankincense (also known as olibanum or, simply, incense) is the name given to the gum-resin harvested from small deciduous trees of the genus *Boswellia* which grow in mountainous regions of eastern Africa (Eritrea, Somalia, Sudan), southern Arabia (Oman, Yemen) and north-west India (Howes 1950; Tucker 1986). Around 20-30 species of *Boswellia* have been described although misidentification and inaccurate nomenclature has created considerable taxonomic confusion, particularly regarding the botanical origin of commercial products (Hamm *et al.* 2003; Thulin and Warfa 1987; Woolley *et al.* 2012). The latter may be derived from a combination of *Boswellia* species' exudates and are often mixed ('cut') with less expensive resins such as pine (Archier and Vieillescazes 2000; van Vuuren *et al.* 2010), a practice of great antiquity (*Dioscorides 1.81* nd; *Pliny NH 12.32:65* nd).

Today, the four main resin-producing species are: *B. carterii* (eastern Africa); *B. frereana* (northern Somalia); *B. sacra* (Arabian Peninsula) and *B. serrata* (NW India/eastern Africa) although some argue that *B. carterii* and *B. sacra* are synonymous and that *B. papyrifera* (eastern Africa) was previously an important source (Mathe *et al.* 2004a; Paul *et al.* 2011; Tucker 1986). Incisions are made in the bark which stimulates the release of a white secretion that solidifies into yellow-brown 'tears' (Mertens *et al.* 2009). These tend to be graded on colour, shape, size, odour and function rather than botanical source and are put to a wide range of uses (Mathe *et al.* 2004b; Moussaieff and Mechoulam 2009; Woolley *et al.* 2012). For example, their volatile fractions are employed in the perfume industry, their HMM terpenic compounds are of interest for their medicinal and preservative properties and the gum-resins themselves are still used as incense (Başar 2005: 108-113, 185-194; Howes 1949: 149-153; Langenheim 2003: 367-368).

Despite this level of exploitation, precise taxonomic relationships within the genus *Boswellia* remain obscure although their chemistry has been extensively investigated (e.g. Başar 2005; Langenheim 2003: 363-367; Tucker 1986). This research has revealed an abundance of mono- and sesquiterpenes (Başar *et al.* 2003; Mertens *et al.* 2009; Woolley *et al.* 2012) and a number of pentacyclic triterpenoids with ursane (dominant), oleanane and lupane (primarily in *B. frereana*) skeletons together with the unique isomers, α- and β-boswellic acid, and their derivatives (Culioli *et al.* 2003; Mathe *et al.* 2004a, 2004b; Proietti *et al.* 1981; **Figure 5.6**).

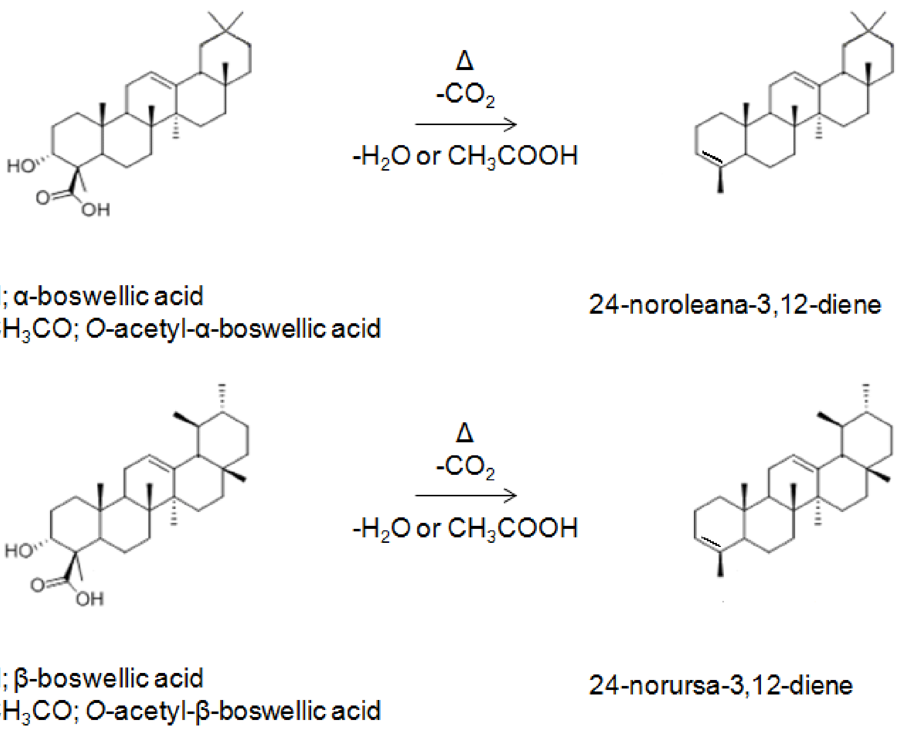


Figure 5.6. Skeletal structures of triterpenoid resin acids diagnostic of *Boswellia* spp. gum-resins: α-boswellic acid (3α-hydroxyolean-12-en-24-oic acid)); β-boswellic acid (3α-hydroxyurs-12-en-23-oic acid); their *O*-acetyl derivatives and principal degradation products (Figure modified from van Bergen *et al.* 1997: 8411).

Table 5.6. Botanical source, geographical spread and chemistry (non-polysaccharide fraction) of gum-resins available and/or attested within the Roman Empire.

Product(s)/taxonomy	**Occurrence**	**Chemistry - phenolics and terpenoids**	**References**
Frankincense/olibanum Family: Burseraceae Genus: *Boswellia* spp.	Africa, Asia, the Americas East Africa, southern Arabia and NW India	Monoterpenic compounds: e.g. α- and β-pinene, sabinene, limonene Sesquiterpenic compounds: e.g. β-caryophyllene Diterpenic compounds: e.g. incensol, serratol and verticilla-4(20),7,11-triene Triterpenic compounds: tetracyclic tirucallanes and dammaranes: e.g. tirucallol, β-elemonic and β-elemolic acid; pentacyclic ursanes (dominant), oleananes and lupanes (primarily in *B. frereana*) e.g. α- and β-amyrin, lupeol, the unique α- and β-boswellic acids and their derivatives	Başar 2005; *et al.* 2001 Culioli *et al.* 2003 Fattorusso *et al.* 1985 Mertens *et al.* 2009 Proietti *et al.* 1981 van Vuuren *et al.* 2010
Myrrh Family: Burseraceae Genus: *Commiphora* spp.	Africa, Asia, the Americas Southern/eastern Africa, southern Arabia and India	Monoterpenic compounds: e.g. α- and β-pinene Sesquiterpenic compounds: e.g. β-elemene, β-selinene, δ-cadinene Furanosesquiterpenes: e.g. curzerene, germacrene derivatives, lindestrene Triterpenic compounds: dammaranes, ursanes and oleanane skeletons e.g. α- and β-amyrin, the commic acids and their derivatives	Bleton and Tchapla 2009 Dekebo *et al.* 2002a, 2002b Hamm *et al.* 2004 Thomas 1961

Low levels of tetracyclic triterpenoids (e.g. tirucallol, β-elemonic acid) and/or cembrene-based diterpenic compounds (e.g. incensol, serratol) have also been detected in some species (Başar *et al.* 2001; Fattorusso *et al.* 1985; Hamm *et al.* 2005; **Figure 5.7**). This production of both di- and triterpenic moieties by a single genus is highly unusual and appears to be restricted to *Boswellia* spp. exudates with the main diterpenic compound, incensol, of relatively rare occurrence (Hamm *et al.* 2003).

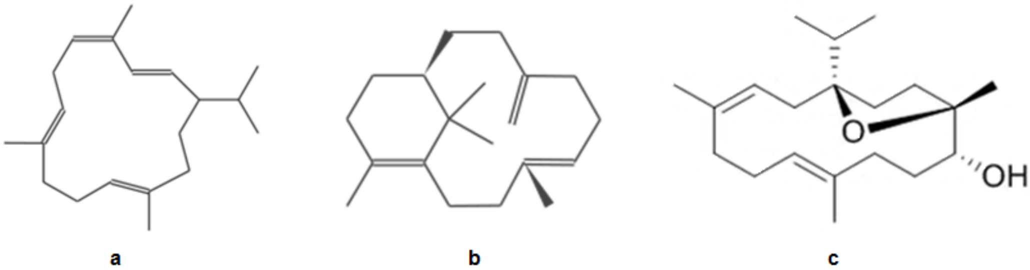


Figure 5.7. Skeletal structures of diterpenic compounds in *Boswellia* spp. gum-resins: a. cembrene C (1,7,11-trimethyl-4(propanyl)cyclotetradecatetraene); b. verticilla-4(20),7,11-triene; c. incensol (1,5,9-trimethyl-12-propan-2-yl-15-oxadicyclopentadeca-5-9-dien-2-ol) (Author, structures from NIST 2016).

Nonetheless, it is the presence of the boswellic acids (often accompanied by their acetylated derivatives) that has long been deemed necessary in order to provide definitive evidence for frankincense. Recent research has shown, however, that these key biomarkers may not be abundant in all species (e.g. *B. frereana*) and that they are preferentially degraded as a result of pyrolysis (Başar 2005: 147-148; Baeten *et al.* 2014; Mathe *et al.* 2007). Thus, low levels or the absence of these resin acids and a significant increase in their degradation products (e.g. 24-noroleana-3,12-diene, 24-norursa-3,12-diene; **Figure 5.6)** may be indicative of heating (i.e. burning as incense) prior to deposition with the triterpenic alcohols, their epimers and derivatives less affected by thermal decomposition (Başar 2005: 151-184; Mathe *et al.* 2007; van Bergen *et al.* 1997; **Figure 5.8; Table 5.7**). What remains unclear, is whether or not natural environmental interactions result in similar diagenetic changes (which, as demonstrated above, is the case for other exudates) since this aspect of *Boswellia* spp. chemistry has rarely been evaluated (Mathe *et al.* 2004a; Regert *et al.* 2008).

Table 5.7. Comparison of triterpenic biomarkers reported in the literature as present in *Boswellia* spp. (compiled from Başar 2005; Culioli *et al.* 2003; Hamm *et al.* 2005; Mathe *et al.* 2004a, 2007; Mertens *et al.* 2009; Paul *et al.* 2011; Proietti *et al.* 1981). X denotes presence; *† B. carterii and B. serrata; ‡ 'Eritrean'; * after pyrolysis; ** B. serrata only*. Roman numerals relate to **Figure 5.8**.

Name of compound	***B. carterii* (E. Africa)**	***B. sacra* (Arabia)**	***B. serrata* (India)**	***B. frereana* (Somalia)**	**Pyrolysates† (Başar 2005)**	**Mathe *et al.* 2007‡**	
						Fresh	**Heated**
24-norolean-3,9(11),12-triene	*X**		*X**		X		
24-norursa-3,9(11),12-triene	*X**		*X**		X		
24-norolean-3,12-diene	X		X		X		I
24-norursa-3,12-diene	X		X		X		II
3-*epi*-β-amyrin	X	X	X	X		IV	IV
3-*epi*-α-amyrin	X	X	X	X		V	V
3-*epi*-lupeol	trace	trace		MAIN		VI	VI
24-norursa-3,12-dien-11-one	*X**		*X**		X		
β-amyrenone	X	X	X	X	*X***	VII	VII
β-amyrin	X	X	X	X	*X***	VIII	VIII
α-amyrenone	X	X	X	X	*X***	IX	IX
α-amyrin	X	X	X	X	*X***	X	X
lupenone	trace	trace		X			
lupeol	trace	trace	trace	X			
α-boswellic acid	X	X	X	trace		XIII	
β-boswellic acid	MAIN	MAIN	MAIN	trace		XIV	
lupeolic acid	minor	minor	minor			XV	
3-oxo-8,24-tetradehydrotirucallic acid	X	X	X	trace	trace		
3-*O*-acetyl-8,24-tetradehydrotirucallic acid	X		X		trace		
3-*O*-acetoxy-9,11-dehydro-β-boswellic acid	trace		trace				
3-*O*-acetyl-α-boswellic acid	X	X	X			XVI	
3-*O*-acetyl-β-boswellic acid	X	X	X			XVII	
3α-*O*-acetyl-lup-20(29)-en-24-oic acid	X	X	X			XVIII	
11-keto-β-boswellic acid	X	X	X	trace			
3-*O*-acetyl-11-keto-β-boswellic acid	X	X	X				

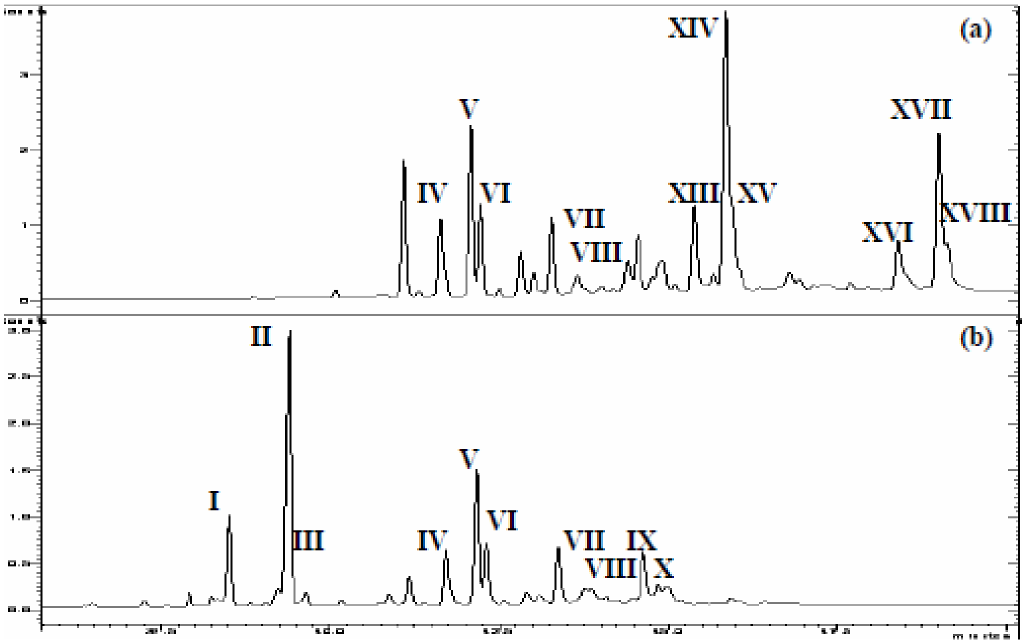


Figure 5.8. The effects of thermal degradation on *Boswellia* spp. resin; a. fresh resin, b. heated resin (Figure from Mathe *et al.* 2007: 440). For peak identifiers, see **Table 5.7**.

It may be, therefore, that the use of *Boswellia* spp. resins in the past predominantly as incense accounts for their infrequent discovery in the archaeological record given their seemingly widespread significance and utilisation (Serpico 2000; Tucker 1986; **3.2.2**). The earliest literary reference is found in Herodotus who identifies southern Arabia as the source of frankincense in the 5th century BC (*Thalia 3.107-110* nd). This is followed by Theophrastus (4th century BC) who provides details regarding the collection of this highly valued exudate from small trees growing in a mountainous area controlled by the Sabaeans (*9.4* nd). This region, known as the 'Frankincense Kingdom' was termed 'blessed' on account of its botanical riches (*Pliny NH 12.30-32* nd) and remained the main focus of the trade in the Roman period (*Anon PME 20-32* nd). Chemical confirmation for this level of exploitation of frankincense has, however, been scarce. In fact, prior to this project, only a handful of published studies were able to demonstrate its presence at sites in Egypt (Mathe *et al.* 2004a), Nubia (Evershed *et al.* 1997a) and Yemen (Mathe *et al.* 2007; Regert *et al.* 2008; **Table 5.8**).

The first of these finds came from the analysis of resinous materials recovered from a house cellar in the frontier settlement of Qasr Ibrîm, Nubia dated c. AD 400-500 (Evershed *et al.* 1997a; van Bergen *et al.* 1997).

Identification of the α- and β-boswellic acids, their corresponding *O*-acetyl and 24-nordiene derivatives demonstrated that a small number of the fragments sampled represented frankincense, with the remainder found to be Pinaceae exudates. As neither members of the Pinaceae nor *Boswellia* spp. grow in northern Africa these resins must have been imported. Likewise, the earliest example, dated 1897-1844 BC, also pertained to an Egyptian context (Mathe *et al.* 2004a). This comprised a black amorphous mass recovered from an ointment jar placed within a scent casket as part of the funerary equipment of Sat-mer-Hout, sister of Amenemhat I (12th Dynasty), Dahshour, Egypt. Again, the α- and β-boswellic acids together with lupeolic acid and their derivatives enabled the authors to identify aged or thermally degraded frankincense in this Ancient Egyptian unguent. Traces of abietane, pimarane and oxidised abietic acid derivatives characteristic of aged and/or heated Pinaceae resins and carboxylic acids, possibly indicative of a vegetable oil, were also present.

Two other discoveries pertain to ancient and medieval Yemen, the region of southern Arabia which in antiquity (as today) was a major source of frankincense (*Anon PME 27-32* nd; Mertens *et al.* 2009; *Pliny NH 12.30-32* nd). Resinous samples from a warehouse and the central sanctuary at ancient Qana' (1st-5th century AD) were analysed and found to contain *Boswellia* spp. biomarkers (Mathe *et al.* 2007). Thermal degradation experiments on modern materials (**Figure 5.8**) and analysis of the archaeological samples using geochemical techniques as well as GC-MS showed that the resin fragment from the warehouse was relatively well preserved. The samples from the sanctuary, two of which were from incense burners, were considerably more degraded with traces of acid components in the white powdery sample but not in the dark amorphous residues which had almost certainly been heated. Likewise, analysis of translucent substances from the 10th -11th century AD medieval site of Sharma, Hadramawt, Yemen proved, in a small number of cases, to be frankincense although the majority contained a range of diterpenoids probably indicative of *Hymenaea* spp. resins, East African copals (Regert *et al.* 2008).

Table 5.8. Summary of previous chemical identifications of frankincense in the archaeological record.

Site/Date	**Sample(s)**	**Results**	**Interpretation**	**Reference**
Dahshour **Egypt** 1897-1844 BC (12^{th} Dynasty)	Black amorphous mass obtained from an ointment jar placed within a scent casket. Part of the funerary equipment of Sat-mer-Hout, sister of Amenemhat I.	1. α- and β-boswellic acids, lupeolic acid, *O*-acetyl and 24-nor-diene derivatives. 2. Trace levels of abietane, pimarane and oxidised abietic acid derivatives. 3. Carboxylic acids.	Mixture of aged or thermally degraded frankincense, aged and/or heated Pinaceae resin and ?vegetable oil.	Mathe *et al.* 2004a
Port of Qana' **Yemen** Late 1^{st}-early 5^{th} c. AD	Resinous fragment from a burnt warehouse. Powder from incense burners and amorphous masses found in the sanctuary of a temple.	1. α- and β-boswellic acids, lupeolic acid and their *O*-acetyl derivatives	Aged frankincense from the warehouse. Aged and/or thermally degraded frankincense from the temple.	Mathe *et al.* 2007
Naintré **Poitou-Charentes** **France** 3^{rd} c. AD	Residues from 2 sarcophagi with lead liners from vaulted tombs. Burials of an adult female and c. 12 year old child.	1. α- and β-boswellic acids and their *O*-acetyl derivatives 2. moronic, oleanonic, masticadienonic and *iso*masticadienonic acids	Frankincense and *Pistacia* spp. resins on and around both bodies. Black substance interpreted as *Boswellia* spp. bark located above the remains.	Devièse 2008
Qasr Ibrîm **Nubia** 400-500 AD	Resinous fragments recovered during sieving of floor surface of house cellar	1. α- and β-boswellic acids, *O*-acetyl and 24-nor-diene derivatives 2. diterpenic compounds, including *iso*pimaric, abietic and dehydroabietic acid	Small percentage of aged or thermally degraded frankincense. Larger percentage of Pinaceae resin fragments.	Evershed *et al.* 1997a van Bergen *et al.* 1997
Wallonia **Southern Belgium** 12^{th}-14^{th} c. AD	Residues with charcoal within perforated ceramic vessels (re-used domestic wares) deposited in medieval mortuary contexts associated with ecclesiastical buildings.	1. α- and β-boswellic acids, lupeolic acid, tirucallic acids and their derivatives 2. cembrane-skeleton diterpenic compounds e.g. incense and serratol 3. diterpenic compounds e.g. pimaric, *iso*pimaric and sandaracopimaric acid with abietane derivatives e.g. dehydroabietic acid and retene 4. phenolics e.g. ferruginol and totarol	Aged and probably mildly heated frankincense mixed with Cupressaceae and Pinaceae products (?juniper and ?pine tar). Burnt as incense with charcoal as an accelerant within perforated ceramics used as censors.	Baeten *et al.* 2014
Sharma **Hadramawt** **Yemen** 10^{th}-11^{th} c. AD	Resinous fragments associated with buildings and ceramics from all levels of the site, a medieval port.	1. diterpenic compounds including ozic acid 2. α- and β-boswellic acids	A small percentage of frankincense among many diterpenoid-containing fragments, probably *Hymenaea* spp. (copal).	Regert *et al.* 2008

Any identification of ancient frankincense is significant but its presence in these regions is not altogether unexpected given the geographical spread of the genus and the known commercial distribution of its exudates. Subsequent research has, however, revealed molecular evidence of frankincense in incense burners from medieval (12^{th}-14^{th} century AD) mortuary contexts in Belgium (Baeten *et al.* 2014) while an unpublished thesis details its presence in lead-lined sarcophagus burials from Roman Gaul, in conjunction with *Pistacia* spp. resins (Devièse 2008: 115-131; **3.3.4**). The latter is of considerable importance in relation to the current research agenda as aromatic substances from mortuary contexts in Britain were considered by antiquarians to be gum-resins, possibly frankincense (Gage 1934; Waugh 1962; **4.3**).

The use of myrrh in perfumery, for medicinal purposes, as an astringent and in the ritual sphere is also well-attested (*Dioscorides 1.77* nd; Groom 1981: 17-21; Langenheim 2003: 368-373; *Pliny NH 12.33-35* nd; Shen *et al.* 2012; *Theophrastus 9.4* nd; **3.2**). Obtained from a number of *Commiphora* spp. (c. 250) native to Africa, Arabia and India, this viscous yellow-white liquid rapidly hardens to red-brown fragments with a distinctive odour (Howes 1949: 153). As with other exudates, confusion with regards to taxonomy and source prevails with various bdelliums or balsams believed to derive from *Commiphora* spp. (e.g. opopanax) (Baumann 1960; Tucker 1986). The secretions from this diverse genus are also difficult to identify in the archaeological record as they are mainly composed of water-soluble, non-diagnostic polysaccharides (<60%) and LMM volatile components (c. 8%) (Hamm *et al.* 2004). The latter comprise common mono- and sesquiterpenes (e.g. α-pinene, β-elemene, α-copaene, β-selinene, δ-cadinene) and more unusual furanosesquiterpenes (e.g. curzerene, germacrene derivatives, furanodienes, lindestrene) (Bleton and Tchapla 2009; Dekebo *et al.* 2002a).

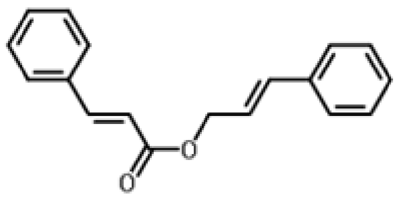
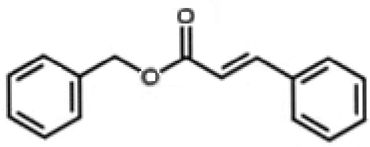
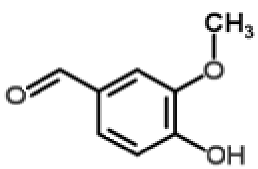
In addition, triterpenic compounds with dammarane, ursane and oleanane skeletons (e.g. α- and β-amyrin and commic acids) have been identified in species of limited geographical spread e.g. *C. pyracanthoides,* southern Africa and *C. confusa*, eastern Africa (Dekebo *et al.* 2002b; Hanuš *et al.*

2005; Shen *et al.* 2012; Thomas 1961). Thus, chemical indications for myrrh, even in well-preserved archaeological materials, are rare (e.g. Buckley *et al.* 2004; Vieillescazes and Coen 1993; Serpico 1996: 125-139, 227-229) despite being listed as a key ingredient of Ancient Egyptian embalming mixtures (Baumann 1960) and seemingly employed in Roman period mortuary rites (**3.2.2**).

5.5 Balsamic resins

Balsamic resins also contain triterpenoids but are mainly composed of free and bound phenolic acids (e.g. benzoic acid, cinnamic acid) with the latter often esterified with various aryl alcohols (e.g. cinnamyl, coniferyl, *p*-coumaryl alcohol) (Colombini and Modugno 2009; Mills and White 1999: 109; **Table 5.9**).

Table 5.9. Key phenolic compounds present in balsamic resins. Chemical structures obtained from RSC (2015); TMS = trimethylsilyl derivatives.

Common name IUPAC name	**Chemical structure**	**Formula** **Mass**
Cinnamyl cinnamate 3-phenyl-2-propen-1-yl-3-phenylacrylate		$C_{18}H_{16}O_2$ 264 TMS NA
Benzyl cinnamate benzyl-3-phenylprop-2-enoate		$C_{16}H_{14}O_2$ 238 TMS NA
Cinnamic acid 3-phenylacrylic acid		$C_9H_8O_2$ 148 TMS 220
Vanillin 4-hydroxy-3-methoxybenzaldehyde		$C_8H_8O_3$ 152 TMS 224

Those of interest comprise the exudates known in antiquity as styrax and storax whose relationship and botanical sources are obscure (Serpico 1996: 58-60; **Table 5.10**). Indeed, these terms are often used interchangeably (e.g. Baumann 1960; Guenther 1943; Langenheim 1996) and it has even been suggested that styrax may be purified storax (Wallis 1967) or that one denotes a liquid (styrax) and the other a solidified (storax) exudate, although probably from different genera (Lardos *et al.* 2011). In the classical texts, references are scant and equally ambiguous with Theophrastus simply listing storax among the "*αρώματα*" (aromatics; *9.7:3* nd) while Dioscorides details a range of medicinal applications for styrax, with the best quality balsam described as "*yellow, fat [and] full of rosin*" (*1.71:2* nd). Obtained from quince-like trees native to the Levant, Greek Islands and Turkish mainland, styrax from Syria was also used in perfumery and for its aromatic wood, according to Pliny (*NH 12.41, 12.55* nd).

Today, storax is the term generally applied to the fragrant balsam derived from members of the genus *Liquidambar* (Altingiaceae, formerly Hammamelidaceae) (Langenheim 2003: 347-348). This resinous substance can be obtained by bruising, scraping and/or stripping the bark/sapwood from these medium-sized trees, boiling it in water and pressing out or skimming off the extract (Guenther 1943; Serpico 1996: 59-60; Wallis 1967). Only one species, *Liquidambar orientalis,* which grows in western Turkey and the Levant would, however, have been accessible during the Roman period (Howes 1950) and could easily be the *"Syrian resin [which] has a resemblance to Attic honey"* described by Pliny (*NH 14.25:123* nd). Likewise, *Styrax officinalis*, the potential source of styrax is the sole representative of the Styracaceae in the Mediterranean and the Levant (Howes 1949: 167). In South-East Asia, other *Styrax* spp. readily exude balsamic resins (referred to as benzoe/benzoin) but modern attempts to acquire an extract from *S. officinalis* have repeatedly failed with (commercial?) collection last reported in the 1700s (Pastorova *et al.* 1997; Serpico 1996: 58-59).

There has, therefore, been much debate over the viability of *S. officinalis* as the ancient source of styrax (Lardos *et al.* 2011). Current explanations favour an apparent decline in productivity due to environmental change (*S. officinalis* is on the red list of endangered species) and/or loss of interest in its cultivation with increased access to cinnamon/cassia as an alternative source of cinnamates, exacerbated by the low yield and labour-intensive nature of styrax extraction (Howes 1950; Langenheim 2003: 355). A reduction in the demand for storax has also been observed (Howes 1949: 162-163) although it continues to play a role in traditional medicine due to its antiseptic properties, in perfumery (primarily as a fixative) and as part of the incense used by the Catholic Church (Baeten *et al.* 2014; Hafizoğlu *et al.* 1996; Pastorova *et al.* 1998; SCCP 2005).

Thus, these species have received relatively little attention with characterisation of balsams from *S. officinalis* reliant, perforce, on the analysis of SE Asian *Styrax* spp. exudates (Hovaneissian *et al.* 2008; Modugno *et al.* 2006a; Pastorova *et al.* 1997). Identification in the archaeological record is further hampered by the fact that the LMM compounds are subject to rapid evaporation, highly variable in nature (qualitatively and quantitatively) and as the result of collection methods and are found in the tissues, extracts and exudates of other plants (e.g. cinnamon, cassia) (Colombini and Modugno 2009; Hovaneissian *et al.* 2008; Leela 2008; Wallis 1967). The research that has been undertaken indicates, with regards to *L. orientalis*, that the major compounds comprise cinnamate esters (e.g. cinnamyl cinnamate, benzyl cinnamate, 3-phenylpropanyl cinnamate) together with free phenolics (e.g. cinnamic acid, benzoic acid, vanillin, cinnamyl alcohol, 3-phenylpropanol) (Guenther 1943; Hafizoğlu 1982, *et al.* 1996; Lawrence 2007; Wallis 1967). The exudates from SE Asian *Styrax* spp. (modern benzoe balsams) contain a similar range of phenolic compounds (e.g. benzoic acid, cinnamic acid, vanillin, cinnamyl alcohol, cinnamyl cinnamate) alongside benzoate and cinnamate esters with aryl alcohols (e.g. *p*-coumaryl, coniferyl alcohol) (Pastorova *et al.* 1997).

A triterpenic contribution has also been noted in *L. orientalis* (Huneck 1963). Generally reported as oleanonic acid and 3α-*epi*oleanolic acid, variations in elution order and mass spectral fragment abundances may indicate the presence of its more common 3β-epimer, oleanolic acid (Modugno *et al.* 2006a; Pastorova *et al.* 1998; see **7.5** for confirmation of the compounds present in *L. orientalis* extracts and their structures). Likewise, oleanonic acid, oleanolic acid (?3β-epimer) and a number of derivatives (alongside lupeol in a potentially adulterated commercial sample) have been identified in balsams obtained from various species of *Styrax* (Hovaneissian *et al.* 2008). Steam distillates (essential oils of low yield) have additionally been investigated. Those from *L. orientalis* (balsam and leaf oils) display a monoterpenic and sesquiterpenic fraction (e.g. α-pinene, limonene, sabinene, α-terpineol, β-caryophyllene) (Duru *et al.* 2002; Fernandez *et al.* 2005; Lawrence 2007) while the leaf oil of *S. officinalis* contains 2-hexenal, octanol and geraniol and its seed oil comprises ubiquitous carboxylic acids (Tayoub *et al.* 2006; Vardar and Oflas 1973).

Despite these challenges, given suitable preservation conditions, balsamic resins clearly have the potential to be identifiable in the archaeological record and warrant further chemical characterisation (**Table 5.11**). This is demonstrated by the discovery of mono- and sesquiterpenes together with cinnamic acid and triterpenes (e.g. oleanonic acid) indicative of a balsamic resin or combination of exudates/extracts in storage jars from the Middle Bronze Age palace at Tel Kabri, Canaan (Israel) (Koh *et al.* 2014). Indeed, the survival of phenolic acids and related compounds including cinnamates, often alongside triterpenic components, in balms from Greco-Roman Egyptian mummies (Buckley and Evershed 2001; Lucas and Harris 1962: 323; Méjanelle *et al.* 1997), residues from an incense burner from the Roman/post-Roman necropolis at Antinoe, Egypt (Modugno *et al.* 2006a), samples from an elaborate lead-lined Roman-style sarcophagus burial from Thessaloniki, Greece (Papageorgopoulou *et al.* 2009) and in similar finds from Rome (Mitschke and gen. Schieck 2012) and Trier (Reifarth 2013: 96-66; **3.3**) appear to indicate that styrax/storax could be of some significance in the mortuary sphere as hinted at by references to the use of balsam in the primary sources (**3.2**).

Table 5.10. Botanical source, geographical spread and chemistry of balsamic resins available and/or attested within the Roman Empire.

Product(s)/taxonomy	**Occurrence**	**Chemistry – phenolics and terpenoids**	**References**
Storax Family: Altingiaceae Genus: *Liquidambar* Species: *L. orientalis*	SE Asia, Mediterranean, Levant, the Americas Mediterranean/Levant	Monoterpenic compounds: e.g. α-pinene, limonene, sabinene, α-terpineol Sesquiterpenic compounds: e.g. β-caryophyllene Phenolic compounds: benzoate and cinnamate esters e.g. cinnamyl cinnamate, benzyl cinnamate, 3-phenylpropanyl cinnamate; free e.g. cinnamic acid, benzoic acid, vanillin, cinnamyl alcohol, 3-phenylpropanol Triterpenic compounds: e.g. oleanonic acid, 3α-*epi*oleanolic acid and isomers	Guenther 1943 Hafizoğlu 1982, *et al.* 1996 Huneck 1963 Lawrence 2007 Modugno *et al.* 2006a Pastorova *et al.* 1998
Styrax Family: Styracaceae Genus: *Styrax* Species: *S. officinalis*	SE Asia, Mediterranean, Levant, the Americas Mediterranean/Levant	Phenolic compounds: benzoate and cinnamate esters e.g. cinnamyl cinnamate, *p*-coumaryl benzoate, coniferyl cinnamate; free e.g. cinnamic acid, benzoic acid, vanillin, cinnamyl alcohol Triterpenic compounds: e.g. oleanonic acid, oleanolic acid and derivatives	Hovaneissian *et al.* 2008 Pastorova *et al.* 1997

Table 5.11. Summary of potential chemical identifications of balsamic resins in the archaeological record.

Site/Date	**Sample(s)**	**Results**	**Interpretation**	**Reference**
Tel Kabri, Canaan Middle Bronze Age	Residues from storage jars	Mono- and sesquiterpenes, cinnamic acid, oleanolic acid	Honey, storax resin or cinnamon as additive to wine	Koh *et al.* 2014
Egyptian mummies c. 2000 BC – AD 400	Embalming materials	1. Phenolics e.g. *p*-hydroxybenzoic acid 2. Benzoic acid 3. Hydroxyhydrocinnamic acid, vanillic acid	1. Plant product, possibly a balsamic resin 2. Storax or benzoin (styrax) balsam 3. Cinnamon/cassia, storax or benzoin balsam or propolis mixed with beeswax	1. Buckley and Evershed 2001 2. Lucas and Harris 1962: 323 3. Méjanelle *et al.* 1997
Necropolis, Antinoe, Egypt 5^{th}-7^{th} century AD	Residue from incense burner	Phenolics e.g. cinnamic acid, vanillic acid, benzoic acid and derivatives Di- and triterpenoids (e.g. oleananes)	Balsamic resin possibly *S. officinalis* Pinaceae resin and *Pistacia* spp. resin	Modugno *et al.* 2006a
Lead-lined sarcophagus Thessaloniki, Greece 3^{rd}-4^{th} century AD	Residue on hair and bone	Sesquiterpenes, vanillin, cinnamates. Di- and triterpenes (not identified)	Embalming materials including essential oils and possibly balsam from *L. orientalis*	Papageorgopoulou *et al.* 2009
San Sebastian, Rome 2^{nd}-3^{rd} century AD	Residue from sarcophagus	Cinnamic acid, *p*-hydroxycinnamic acid	Balsamic resin	Mitschke and gen. Schieck 2012
Sarcophagus burials Below St Maximin's Trier, Germany	Residues from textiles	Phenolics e.g. cinnamic acid, benzoic acid and derivatives Triterpenoids, oleanane-skeletons	Balsamic resin, possibly from *L. orientalis*	Reifarth 2013: 96-99

5.6 Summary

A broad range of plant exudates and extracts were employed within the Roman Empire in perfumery and as medicines with a more limited palette considered appropriate for use in the ritual sphere. There is, however, little confirmation for their use, outside Egypt, as part of mortuary rites and considerable challenges attend the identification of these scented substances in the archaeological record. These include losses due to solubility in water (e.g. polysaccharides present in gums and gum-resins) and volatility (e.g. LMM phenolics and terpenes indicative of essential oils and balsamic resins) together with the common occurrence and natural variability of many compounds, particularly those of LMM (e.g. mono- and sesquiterpenes) although, within a genus, the relative abundance/presence of HMM biomarkers may also differ considerably (e.g. between various *Pistacia* spp. and *Boswellia* spp. exudates, see **Appendix 3**). In addition, the multitude of environmental factors affecting the degradation of the more diagnostic di- and triterpenic components and the potential impact of anthropogenic modification prior to deposition (e.g. extraction methods, mixing, heating) may serve to obscure comparisons with modern reference samples. Issues regarding the relationship between colloquial or trade names (ancient and modern) and botanical sources, changes in geographical spread and/or resin production over time or even the extinction of previously utilised species adds to these difficulties.

Nonetheless, significant progress has been made in this field through interrogation of the phytochemical literature where research into terpenic compounds is ongoing, often due to their medicinal potential. This has been supplemented by projects using multi-analytical approaches to investigate amorphous organic residues from a variety of archaeological contexts in conjunction with experimental degradation studies designed to address specific resin-related questions. Thus, a considerable compendium of knowledge on which to base botanical assignments exists although, inevitably, some genera will be more readily characterised than others. With regards to the Roman period, much of the chemical evidence obtained has indicated the use of Pinaceae resins alongside mastic/terebinth (*Pistacia* spp.), frankincense (*Boswellia* spp.), and, potentially, balsamic extracts in mortuary contexts. Other substances such as essential oils (e.g. aloes, cinnamon/cassia, nard) and exudates like myrrh, which were seemingly employed as part of Roman funerary rites, still await secure identification in the archaeological record.

Based on these observations, when investigating mortuary contexts in Roman Britain, suites of di- and triterpenic compounds are required in order to confirm the presence of an imported resin (Research Question 1 (RQ1); **8.3**). Consideration of the diagnostic resin acids, their derivatives and any surviving LMM components should then enable identification of botanical source, meeting Research Question 2 (RQ2; **8.4**). Where characteristic HMM terpenoids are not present, evaluation of their degradation products may provide indications as to source, although probably at a higher taxonomic level. Tracing these degradation pathways may also enable the question of environmental and/or anthropogenic impacts on the substances employed and the purpose of this practice to be addressed (RQ3; **8.5**) and help build a picture of how this specific practice relates to mortuary rites in Britain (RQ4; **8.6**) and elsewhere in the Roman Empire (RQ5; **8.7**). These findings can then be integrated with mortuary theory to consider the meaning encapsulated in the materiality of Roman death (RQ6; **8.8**). Of course, to begin this process, an appropriate analytical protocol is required.

Chapter 6

Experimental: materials and methods

"In the demon-haunted world that we inhabit...[the scientific method] may be all that stands between us and the enveloping darkness".
The demon-haunted world, Carl Sagan 1997: 408

6.1 Introduction

Plant exudates are complex substances composed of a range of compounds of varying solubility and volatility. Their recovery from archaeological mortuary contexts requires consideration of a number of factors. These include the collection of appropriate samples (**6.2**), determination of the optimum instrumental method(s) of analysis and evaluation any pre-treatment procedures in terms of their impact on the empirical data obtained (**6.3; 6.4**). Contextualisation of any positive results through appraisal of the taxa available during the period and in the region under investigation (**3.2; 3.3**) together with knowledge of the original chemical composition of their exudates is also crucial (**Chapter 5**). Moreover, the possibility that these substances may have formed part of a mixture or been heated in antiquity must be addressed alongside issues of subsequent chemical alteration and contamination as a result of interactions within the burial environment. Thus, evaluation of the source of other extracted lipid components, the analysis of control samples and some understanding of the degradation pathways of resin acids is required. These parameters can be established through comparison with modern reference materials (**6.5**), authentic and synthesised standards (**6.6**) and the published literature (**Chapter 5**). Targeted degradation studies also have a significant role to play in tracing taphonomic processes but did not form part of this research agenda.

In response to the considerable analytical challenge posed by these materials, a range of instrumental techniques were employed. These fall into two categories: non-destructive spectroscopic methods such as Fourier transform infrared (FTIR; **6.3.1**) and/or Raman (**6.3.2**) spectroscopy and minimally-destructive chromatographic and spectrometric methods, the most effective being gas chromatography-mass spectrometry (GC-MS; **6.4**)

(Evershed 2008a; Modugno and Ribechini 2009). The spectroscopic methods have been shown to be useful as rapid, first pass techniques which permit the general category of a substance to be determined due to characteristic vibrational frequencies of the chemical bonds and functional groups present (Pollard *et al.* 2007: 77-85). They are not, however, sufficiently sensitive to permit further secure interpretation of the results especially when considering aged resinous materials within a mixed matrix (Evershed 2008a; Stern *et al.* 2008b). Nonetheless, as destructive techniques are not always appropriate and portable versions of these instruments (which could be used on location in museum stores or during excavation) are becoming increasingly available, their utility was evaluated.

To obtain the necessary level of compositional information, the focus of the analytical work was the application of GC-MS. This technique has become the method of choice for detailed chemical characterisation of organic materials of archaeological interest (Evershed 2008a; Modugno and Ribechini 2009). GC-MS enables a broad range of compounds to be separated based on their polarity and molecular mass as the result of interactions between a stationary (fused silica) and mobile (gas) phase, with partitioning possible to the level of isomers (Harris 2007: 528-551). As the compounds elute into the mass spectrometer they are ionised and fragmented in characteristic and reproducible ways which permit their structural identification (Pollard *et al.* 2007: 143-156). Evaluation of botanical source is then, predominantly, based on the presence and relative abundance of suites of the higher molecular mass terpenic moieties which form part of the resin fraction, permitting taxonomic classification, generally to the level of genus (Serpico 2000).

In addition, nuclear magnetic resonance (NMR) spectroscopy and atmospheric pressure chemical ionisation-liquid chromatography-mass spectrometry (APCI-LC-MS) were utilised in the characterisation of a number of triterpenoid standards. These were purchased or synthesised to clarify issues regarding the elution order and fragmentation patterns of key epimeric pairs (**6.6; 7.5**).

After consideration of these factors a protocol was developed detailing the sampling methodology and sequence of analytical processes required to optimise the level of information obtained from the samples (**Figure 6.1**).

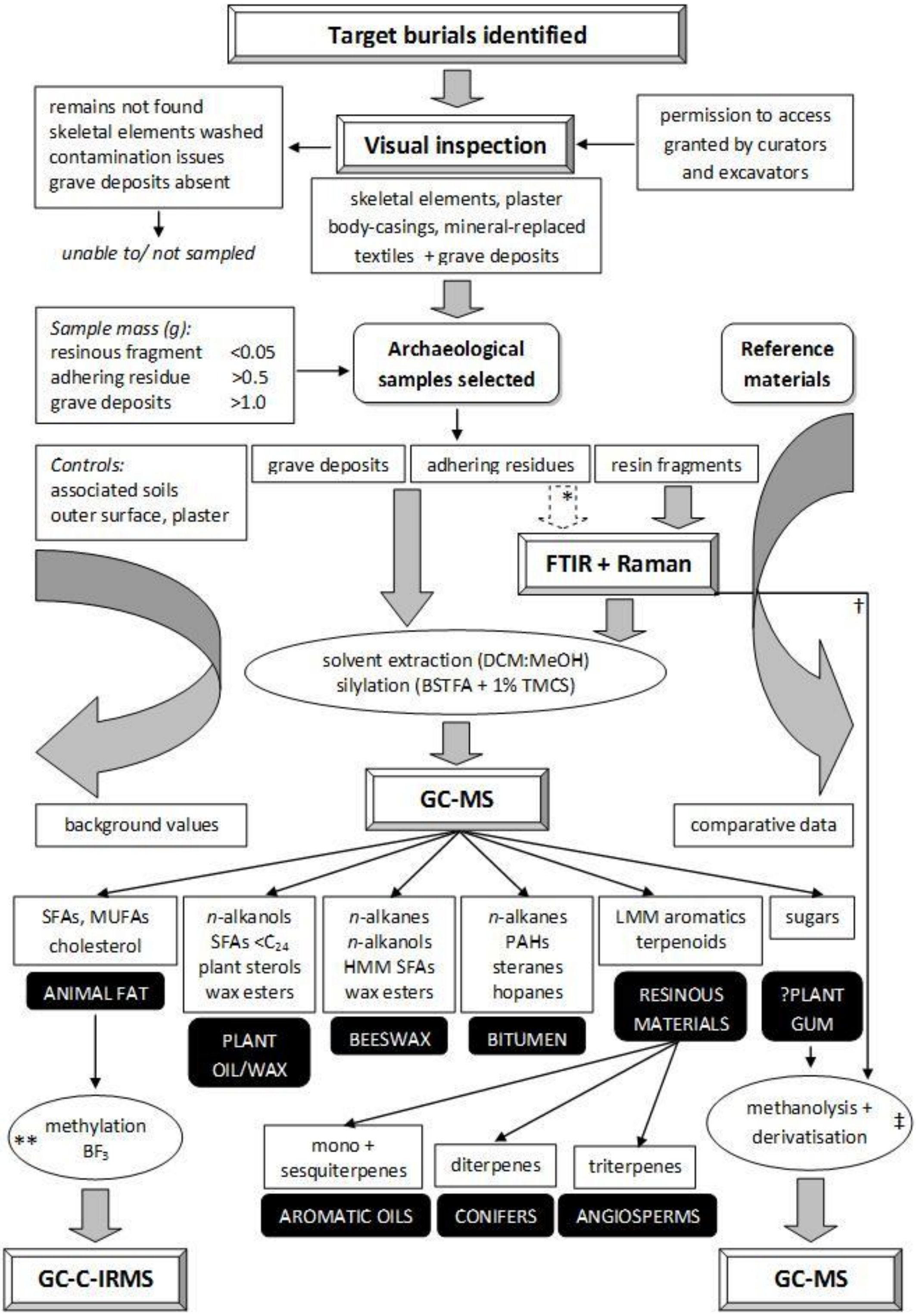


Figure 6.1. Sample protocol produced for this project. *adhering residues could be assessed if portable instruments were available; **if present in sufficient abundance the origin of fats could be determined by GC-C-IRMS (e.g. Evershed 2009); †if saccharides are present these could be investigated by Raman and/or FTIR, ‡with confirmation by GC-MS (e.g. Ménager *et al.* 2014).

6.2 Sample selection

There is an increasing reluctance among museum curators to permit the removal of samples for scientific analysis. This is exacerbated when the materials requested are in any way related to sensitive artefacts such as human remains. Thus, when permission is granted, it is imperative that the impact should be minimal and that sub-sampling should take place whenever possible so that a portion is retained for future research and display, if appropriate. With this in mind, prior to sample collection the minimum mass for the effective analysis of a 'pure' resinous fragment was ascertained. Using a modern reference (*Pistacia* spp.) resin, samples of decreasing mass were analysed and the minimum amount required empirically determined to be ~0.005 g. In terms of more friable and degraded archaeological materials, however, it was considered advisable to recommend collection of a ~0.05 g sample to enable a range of complementary techniques, comparative derivatisation methods and repeat analyses to be undertaken, if required.

The recovery of resinous fragments is rare in the archaeological mortuary record. Their amorphous nature means that they are easily overlooked during excavation (Evershed *et al.* 1997a) or may have become powdered and essentially invisible (personal observation). Thus, a systematic approach was taken towards the selection of a range of samples with the potential to contain resin biomarkers. Amorphous orange and yellow/white masses together with yellow-brown residues adhering to skeletal elements and plaster body-casings were sampled where visible. Sub-samples of degraded materials, termed 'grave deposits', comprising a mixture of inorganic and organic matter from the base of the burial container, were also collected when extant (**Figure 6.2)**. The context and nature of each sample was recorded on a standardised form and photographs taken (**Appendix 2a**).

In archaeological mortuary contexts, it was clear that any resin-derived compounds would be admixed with matter from a variety of other sources related to initial deposition and subsequent taphonomic factors. These components could derive from degraded body tissues, textiles and associated dye-stuffs, floral tributes and other offerings based on evidence for Roman burial practices (**2.3**) and, in many cases, would include soil ingress. Metal ions and carbonates could also be present, particularly when the individual had been encased in plaster or interred in a lead-lined container. Most samples were, therefore, viewed as similar to archaeological soils while residues obtained from plaster body-casings or mineralised textile fragments were deemed comparable with those adhering to or adsorbed within ceramic matrices. Previous research has shown that 1.0 g is the minimum amount of substrate recommended for the characterisation of lipids associated with ceramics and that at least 2.0 g is required in relation to soil samples (Brettell 2007; Evershed *et al.* 2002). This is not an exact science, however, as the original abundance of any resinous substances deposited, their nature (i.e. terpenoid rich, thermally altered) and subsequent life histories will all impact upon the recovery of data of sufficient quality to enable definitive identification (Evershed 2008b). It was decided, therefore, that residue samples >0.5 g and grave deposits >1.0 g would be analysed.

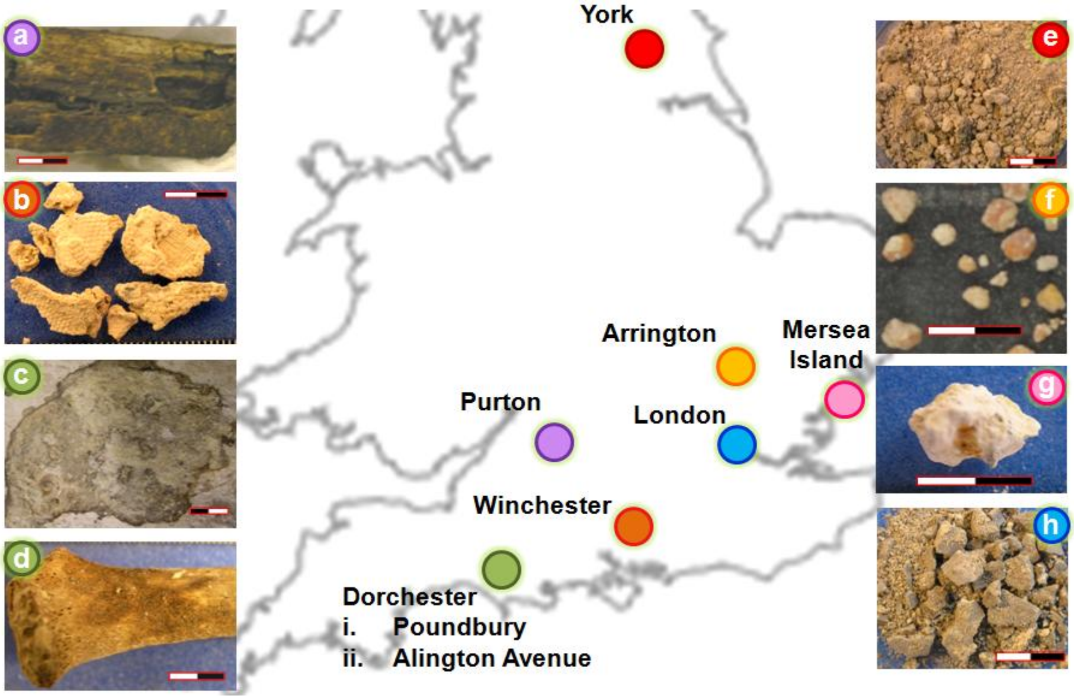


Figure 6.2. Examples of the samples analysed. a. residue-coated femur, lead-lined sarcophagus, Grave 1, Purton, Wiltshire; b. mineral-replaced textiles, lead-lined coffin burial, G336, Eagle Hotel, Winchester; c. dark residue adhering to inner surface of plaster, lead-lined plaster burial, B530, Poundbury, Dorchester; d. residue, humerus, G4378, Alington Avenue, Dorchester; e. grave deposits, base of sarcophagus, plaster burial YORYM:2010.1219, York; f. resinous fragments, lead-lined coffin, infant burial, Arrington, Cambridgeshire; g. amorphous mass, glass cremation urn, Mersea Island barrow, Essex; h. grave deposits, intact lead-lined sarcophagus, SK15903, 'Spitalfields lady', London (Author).

In light of these observations, an ideal sampling strategy was devised based on standard excavation procedures for mortuary contexts (i.e. the use of a horizontal and, where possible, a vertical grid pattern to map the grave) and recent research with regards evaluation of body treatment and factors affecting decomposition in situ (Usai *et al*. 2014) The aim of this approach was to establish background parameters through the selection of controls (i.e. from outside the grave cut and above the container/body to assess the on-site soil profile) and to provide multiple samples of appropriate mass from within each burial (ideally one from each grid square, although constraints of time, money and the abundance/nature of the grave deposits mean that strategic sample selection is probably more realistic in most cases). These samples should be focused on key areas (i.e. head, torso, pelvis, legs/feet, arms/hands), be distributed head to toe, in the mid-line and towards the extremities but also beyond the contours of the body in order to maximise spatial information regarding the distribution (i.e. presence and relative abundance) of any resinous components and their relationship with the human remains. Suggested sample locations, depending on the abundance of grave deposits, are shown in **Appendix 2b**. In practice, body position and other context specific factors will also need to be taken into consideration.

With regards to the current project, these good intentions were severely constrained by methods previously employed during excavation, post-excavation handling and the retention policies of the curatorial museums with date of discovery (1848-2007) a significant factor. Antiquarian practices affecting the burials of interest were found to include the reburial of human remains, sale of lead-liners (sometimes ‘re-packaged' as souvenirs, Hall 1996) and the open-air display of stone sarcophagi. Subsequent losses as a result of misappropriation or misadventure had also occurred (**Table 4.2**). In more recent times, the washing of skeletal elements for osteological analysis and/or display, wet and dry sieving of grave deposits and constraints on the continued curation of bulk samples in museum stores have further impacted on the survival of suitable materials. Phthalate plasticiser contamination was also expected as the majority of the materials sampled had been stored in plastic containers or wrappings. Thus, resin fragments, adhering residues and multiple, spatially-recorded grave deposits were rarely encountered and controls had to be selected from extant materials (e.g. external surface of plaster body-casings, associated soil samples). Only suites of characteristic terpenoids were, therefore, considered representative of the inclusion of resinous exudates.

In total, 195 archaeological samples and controls from Roman period mortuary contexts were analysed as part of this research project (full details are given in the relevant case studies, **Chapter 7**). These comprised:

- materials from 38 inhumations from across Britain in which individuals had been interred in stone sarcophagi, lead-lined coffins and/or with plaster body-casings (**7.2-7.10**);
- organic substances from two, similarly elaborate, multi-container cremation burials from Purton, Wilshire and the Mersea Island barrow, Essex (**7.7;7.11**);
- the solvent-wash of the plait of hair recovered from the lead-lined gypsum burial, Crown Buildings, Dorchester, UK (**7.8**);
- samples from 31 normative inhumation burials (wooden coffin and/or shroud) but where plaster or unusual residues were also indicated.

In addition, to provide further comparative data, soil samples from normative (i.e. supine, extended, in wooden coffins and/or shrouds) inhumations from the burial ground at West Smithfield, London (curated at the Museum of London) were sub-sampled. Twenty-eight of these had been collected from the mid-torso region of the skeletal remains (originally with a view to parasite analysis) and were analysed as part of a pilot study and an undergraduate dissertation. No evidence for the presence of resinous substances was observed in these burials (Harrison *et al.* 2013; Harrison 2014).

This range of burial types was selected to address key objectives regarding associations between the use of plant exudates, presence of plaster, interment in substantial containers and aspects of the deceased’s identity. To supplement the current literature regarding continental finds, four samples from a Roman period sarcophagus from Bezannes, north-eastern France (supplied by Denis Bouquin, Archaeo-Anthropology Scientific Officer, Reims-

Métropole) were also investigated (**Appendix 4**). This plaster burial was of particular interest as exotic pollen from southern Arabian and/or eastern African taxa had recently been recovered (Corbineau 2012, 2014). Finally, in order to address the hypothesis put forward in the 1970s that this body treatment had spread from northern Africa (Ramm 1971; **4.4**) and to counteract the common misconception that any indication of body preservation can be directly equated with Ancient Egyptian mummification practices, seventeen votive mummy bundles (and one sample from a human mummy were analysed; **Appendix 5**). These were included as a proxy (other human mummy samples were not able to be accessed) for considering the nature and pre-application processing of embalming materials utilised in the Greco-Roman period since previous research had indicated similarities between human and non-human mummification processes (Buckley *et al.* 2004; Bruno 2013).

Precise details regarding the location and nature of the samples obtained are discussed in the relevant case studies in **Chapter 7**. These archaeological samples were accessed with the kind permission of the contributing museums facilitated by their curatorial staff with those from the newly-discovered lead-lined coffin from Ilchester obtained by the author during laboratory micro-excavation under the supervision of Robert Croft, County Archaeologist, Somerset (**7.9**). At each institution, a clean working area was prepared and the samples placed within glass scintillation vials for transfer to the Molecular Laboratory, Archaeological Sciences, University of Bradford, Bradford, UK. All glass and metal wares were pre-cleaned and re-cleaned (triple-rinsed in dichloromethane) between each sample, as required.

6.3 Non-destructive analysis

Where visible fragments of resinous appearance were collected, a preliminary assessment was undertaken using a non-destructive technique. This comprised either Fourier transform-infrared spectroscopy and/or Fourier transform Raman spectroscopy.

6.3.1 Fourier transform-infrared spectroscopy (FTIR)

Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) permits bulk structural information about the compounds present in the sample to be ascertained based on the vibrational frequencies of covalent bonds possessing dipole moments (Lampman *et al.* 2010: 15-23). Measurements were performed using a PerkinElmer Frontier spectrometer (4000-650 cm^{-1}, 16 scans, 4 cm^{-1} spectral resolution) without sample pre-treatment. The presence of aromatic and/or terpenic components was ascertained through evaluation of diagnostic vibrational group frequencies as reported in the literature (Colombini *et al.* 2009; Derrick *et al.* 1999: 82-108; Ménager *et al.* 2014). These comprised: strong ν(C=O) stretching (ν) bands, ~1720-1690 cm^{-1}; a shifted broad ν(O-H) band ~3450-3300 cm^{-1}; ν(C-O) bands between 1280-1230 cm^{-1}, generally ~1240 cm^{-1}; CH_2 and CH_3 group bending (δ) at 1460-1450/1377-1385 cm^{-1} and rocking (ρ) modes ~730-740 cm^{-1}. In relation to the votive mummy bundles, results were compared with data obtained from controls (plain archaeological textiles, modern feathers) in order to ascertain bands produced by the substrates (**Appendix 5.2**).

6.3.2 Fourier transform Raman spectroscopy (FT Raman)

One of the visible, portable, resin samples from Roman mortuary contexts in Britain (**7.2**) was investigated using FT Raman spectroscopy. This technique is complementary to FTIR as it arises from inelastic scattering rather than absorption and so records molecular transitions resulting from changes in polarisability (Kincaid 1986). Resinous materials can prove problematic, however, as broadband fluorescence, which is related to sample colour, often results in poor quality data (Smith and Clark 2004). Following the protocol developed at the University of Bradford (Edwards *et al.* 1996), FT Raman spectra were obtained using a Bruker IFS66 with FRA106 Raman module attachment (Nd^{3+}/YAG near-infrared excitation at 1064 nm; 4 cm^{-1} resolution; 1000 scans accumulation). A nominal laser power of 270 mW was used with a footprint of approximately 100 μm. Laser power was kept to a minimum in order to prevent thermal alteration and sample degradation. Generic assignments were by made focussing on: medium-strong bands ~1600-1670 cm^{-1} characteristic of conjugated ν(C=C) bonds in resins; ν(CH_2, CH_3)

stretching modes, ~2870-2980 cm^{-1}; $\delta(CH_2, CH_3)$ deformations, ~1300-1490 cm^{-1}; and v(CC) ring vibrations and breathing ~1100-1200 and ~700-750 cm^{-1} (Edwards and Falk 1997; Edwards *et al.* 1996; Brody *et al.* 2002). Unfortunately, the failure of this instrument meant that no subsequent work could be undertaken.

6.4 Minimally destructive analysis

The non-destructive methods (**6.3**) require little or no pre-treatment of the samples and provide bulk information regarding the nature of the compounds present. Gas chromatography-mass spectrometry (GC-MS) permits the separation and characterisation of individual compounds but necessitates extensive sample preparation procedures prior to analysis. Thus, determination of the most appropriate approach for evaluating the presence of plant exudates in relation to the nature and size of the samples available from mortuary contexts was essential. The possibility of attendant alteration (e.g. thermal degradation, methylation) of the compounds of interest was also evaluated (Stern *et al.* 2000; van Keulen 2009). The method chosen for this project was based on protocols specifically developed for the analysis of organic residues of archaeological interest and refined over many years by researchers working in the Molecular Laboratory, Archaeological Sciences, University of Bradford (Pollard *et al.* 2007: 305-306). Minor modifications were made by the author during method development on modern reference resins. Details regarding key choices are given below. Standard operating procedures and attendant COSHH forms are listed in **Appendices 2.3-2.4** and can be found on **Disc 1**.

6.4.1 Solvent extraction for GC-MS

The first stage of the process requires chemical extraction of the lipid fraction from the sample matrix and the removal of any inorganic or polymerised materials. A range of organic solvents can be used depending on the compounds targeted. In this instance, a combination of dichloromethane (DCM) and methanol (MeOH) was selected (2:1, v/v). Although DCM alone is effective in accessing terpenic compounds, the addition of MeOH facilitates the extraction of a wider range of lipids of varying polarities (e.g. phenols) which could be informative. It also sequesters water which can otherwise shorten the life of the GC column and hinders some derivatising agents (see below). The samples were allowed to stand (30 min) in ~2 ml of DCM:MeOH with ultrasonication (5 min) and centrifuging at 2000 rpm (5 min) (~ 650 relative centrifugal force (g)) to assist extraction, dissolution and solid/solution separation. This process was repeated three times in order to achieve maximum yield with minimum contamination. The solvent-soluble fractions were combined, concentrated under a stream of nitrogen gas on a warm hotplate (c. 40 °C) and left to stand overnight to evaporate to dryness to yield the total lipid extract (TLE). All solvents used were high performance liquid chromatography (HPLC) grade. [N.B. alkaline hydrolysis with partitioning based on polarity was considered but would have necessitated multiple GC-MS runs for each sample which was not practicable in relation to the aims of the project].

6.4.2 Derivatisation of extracted organic compounds

Before the analysis of complex organic mixtures by GC-MS can be undertaken, the volatility of certain components needs to be promoted while polar functional groups should be masked to facilitate separation and improve peak shape and resolution (Pollard and Heron 2008: 61-62). A variety of derivatisation methods have been employed (e.g. Antolín *et al.* 2008; Pollard *et al.* 2007: 142-143; van Keulen 2009) with the most frequent being silylation with BSTFA (*N,O*-bis(trimethylsilyl)trifluoroacetamide with 1% TMCS (trimethylchlorosilane) or methylation using diazomethane or boron trifluoride (BF_3) with the latter essential if subsequent compound specific isotope analysis is required. Methylation adds a methyl group ($-CH_3$) to unhindered acid functionalities to create stable methyl esters with minimal side reactions. Obtaining reagents for the production of diazomethane is, however, increasingly problematic and the process of generating these compounds is both time-consuming and hazardous (Antolín *et al.* 2008; Fales *et al.* 1973).

Moreover, this method can mimic or increase the abundance of certain compounds. For example, significant levels of methyl dehydroabietate (MDHA) may denote the pyrolysis of resinous Pinaceae woods while it can also be formed by natural degradation processes (Colombini *et al.* 2003). This methyl ester results from the reaction between methanol, released by the heating or oxidative decay of components in the wood, and the diterpenoid acids present. Thus, derivatisation involving methylation makes MDHA an unreliable indicator of thermal alteration and can obscure information about the burial environment (Hjulström *et al.* 2006; **Figure 6.3**). Silylation using BSTFA with 1% TMCS is a safer, simpler method which adds a trimethylsilyl group ($-Si(CH_3)_3$) to both unhindered acid and alcohol functionalities and so reveals the presence of natural/heat-induced methyl esters (Colombini *et al.* 2009). TMS derivatives are, however, far less stable and hydrolyse readily. Undesirable side reactions and incomplete derivatisation may also occur (Antolín *et al.* 2008; Nicholson *et al.* 2011).

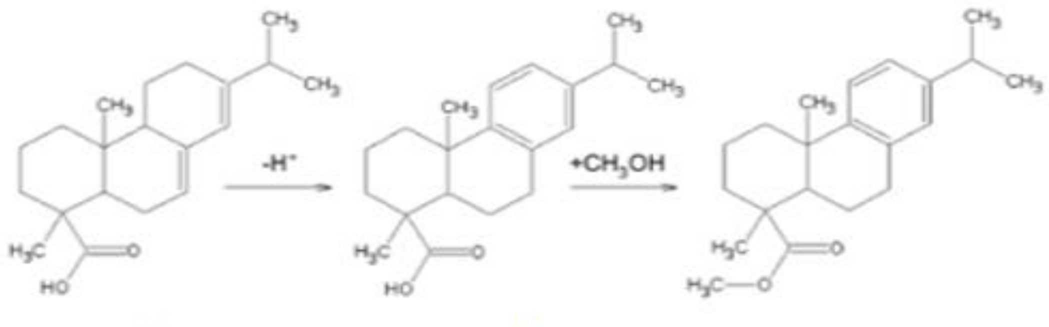


Figure 6.3. Scheme showing abietic acid and main derivatives formed by pyrolysis via dehydrogenation, isomerisation and methylation: a. abietic acid (abieta-7,13-dien-18-oic acid); b. dehydroabietic acid (abieta-8,11,13-trien-18-oic acid); c. methyl dehydroabietate (methyl abieta-8,11,13-trien-18-oate) (Figure from Hjulström *et al.* 2006: Figure 1, 284).

As this project was conceived as a systematic, qualitative evaluation of a large number of samples from Roman period mortuary contexts across Britain, derivatisation using BSTFA with 1% TMCS was considered the most appropriate approach. In addition, much recent research relating to the characterisation of resinous substances has employed silylation (e.g. Baeten *et al.* 2014; Buckley and Evershed 2001; Clark *et al.* 2013; Colombini *et al.* 2003; Hovaneissian *et al.* 2008; Modugno *et al.* 2006b; Regert *et al.* 2008; Stern *et al.* 2008b) and so the production of comparable relative retention time and mass spectral data was deemed prudent. All of the dry total lipid extracts obtained from both the archaeological samples and modern reference resins were, therefore, derivatised using an aliquot (~0.05 ml) of BSTFA with 1% TMCS. Excess reagent was removed by evaporation at room temperature and the derivatised extracts re-diluted with DCM for analysis by GC-MS.

6.4.3 GC-MS conditions

The parameters selected were those developed at the University of Bradford to access a wide range of lipids of differing polarity and mass (Stern *et al.* 2003, 2008b) modified to improve separation through the use of a longer column. These factors were important with regard to the aims of this project as both fresh and aged plant exudates contain a broad range of compounds. In addition, the heterogeneous nature of potential input sources to mortuary contexts required evaluation of components ranging from simple aliphatic molecules such as *n*-alkanes to complex terpenic moieties to aid interpretation. As similar chromatography conditions have been used to good effect in the analysis of resinous substances by other researchers (e.g. Charrié-Duhaut *et al.* 2007; Colombini *et al.* 2005; Modugno *et al.* 2006a; Stacey *et al.* 2006), positive results would also be comparable with the published literature. Data obtained during method development using modern reference materials confirmed the efficacy of this approach.

Analysis was carried out using an Agilent 7890A GC system, fitted with a 30 m x 0.25 mm, 0.25 µm HP-5MS 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple axis mass selective detector. The splitless injector and interface were maintained at 300 °C and 280 °C, respectively, and the carrier gas, helium, at constant flow (1.5 ml/min). The temperature of the oven was programmed to rise from 50 °C (isothermal, 2 min) to 350 °C (isothermal, 10 min) at a gradient of 10 °C per minute. The column was directly inserted into the ion source where electron ionization (EI) spectra were obtained at 70 eV with full scan from *m/z* 50 to 800 amu.

6.4.4 Quality control

Method blanks were prepared with each batch of samples to assess procedural contamination. Solvent blanks (DCM) were incorporated at regular intervals within each run to check for carry over and excessive column degradation. External standards, comprising the in-house microcrystalline wax (a homologous series of *n*-alkanes) and compounds specific to this project (oleanolic acid and ursolic acid) purchased from Sigma-Aldrich and Tokyo Chemical Industry (TCI) UK Ltd., respectively, were analysed at the beginning and end of each run to ascertain chromatographic quality and retention time shifts. The mass spectrometer was calibrated with perfluorotributylamine (PFTBA) using the Autotune function to optimise response and resolution at the start of each batch of samples.

6.4.5 Reporting the GC-MS results

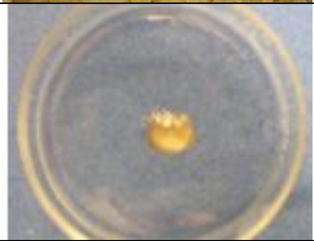
GC-MS results of the silylated solvent extracts of the archaeological and modern samples are presented as total ion current (TIC) chromatograms (often focussing on the elution times of the various terpenoids) and extracted ion current (XIC) chromatograms (which select for suites of related compounds based on their characteristic ion fragments). Each separated component is shown as a discrete peak with the area beneath representative of its relative abundance. Determination was based on the mass of the molecular ion, characteristic fragmentation patterns, peak distribution and retention times of these compounds in comparison with empirical data obtained from the analysis of reference samples (**Appendix 3**), the mass spectral literature and previous phytochemical and archaeological publications. Key components identified and discussed in the text are labelled, with details given in the corresponding tables relating to each Case Study (**Chapter 7**).

6.5 Reference materials

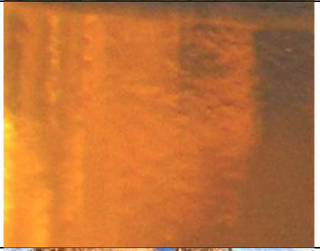
Analysis of plant exudates identified as having been accessible and/or employed in the mortuary sphere within the Roman Empire (**Chapter 3**) was undertaken using the above protocol (**6.1**).

Table 6.1. Details of the modern reference materials selected for comparative analysis. See **Appendix 3** for details of chemistry and chromatograms.

GC-MS code	*Botanical source* **Place of origin**	Common name	*Designation* **Brief description**	Image	Sample provider
CED009	Pinaceae *Cedrus libani* **Levant**	Lebanon cedar	*Diterpenoid resin* Yellow/orange Brittle fragments/powder. Opaque/translucent areas. No scent.		UOB reference collection Reference 009 Royal Botanical Gardens, Kew Reference KEBC 11243
LAR113	Pinaceae *Larix* spp. **Northern Europe**	Larch	*Diterpenoid resin* Yellow/orange. Amorphous fragments. Opaque. No scent		UOB reference collection Reference 113
PIN065	Pinaceae *Pinus pinaster* **France**	Maritime pine	*Diterpenoid resin* Deep orange. Brittle fragments. Opaque. Slight scent.		UOB reference collection Reference 065
NRKFZ	Pinaceae *Pinus sylvestris* **Germany**	Scot’s pine	*Diterpenoid resin* Pale yellow/orange. Angular fragments. Translucent. Slight scent.		Donated sample Nicole Reifarth ‘Colophony’

PST121	Pinaceae *Pinus sylvestris* **Netherlands**	Stockholm tar	*Heated diterpenoid wood tar* Dark brown-black Amorphous viscous masses. Opaque. Powerful aroma.		UOB reference collection Reference 121
PLT012	Anacardiaceae *Pistacia lentiscus* **Greece**	Mastic	*Triterpenoid resin* Pale yellow. Small ‘beads’. Translucent. Slight scent.		Bristol Botanicals Ltd. Reference GAW012
PA	Anacardiaceae *Pistacia lentiscus* **Athens, Greece**	Mastic	*Triterpenoid resin* Pale yellow. Viscous liquid Translucent. Strong scent.		Collected by Dr Ben Stern Summer 2011
PTC055	Anacardiaceae. *Pistacia terebinthus* **Cyprus**	Terebinth	*Triterpenoid resin* Orange/yellow. Amorphous masses. Translucent, some opacity. Slight scent.		UOB reference collection Reference 055

PTP056	Anacardiaceae. *Pistacia terebinthus.* **‘Persia’**	Terebinth	*Triterpenoid resin* Brown/orange. Amorphous masses. Dull, opaque. No scent.		UOB reference collection Reference 056 Royal Botanical Gardens, Kew Reference KEBC 61999
PKI035	Anacardiaceae. *Pistacia khinjuk* **Iran**	-------	*Triterpenoid resin* Orange. Rounded fragments. Largely opaque. No scent.		UOB reference collection Reference 035 Royal Botanical Gardens, Kew Reference KEBC69025
FRKBB050	Burseraceae *Boswellia carterii* **Ethiopia**	Frankincense	*Triterpenoid gum-resin* Yellow-orange/brown. Rounded fragments. Opaque. Strong scent.		Bristol Botanicals Ltd. Reference GAW050 ‘African’
FRKBB052	Burseraceae *Boswellia sacra* **Oman**	Frankincense	*Triterpenoid gum-resin* Pale cream-yellow. Large ‘tears’. Opaque. Strong scent.		Bristol Botanicals Ltd. Reference GAW052 ‘Oman Naeem’

FRKBB009	Burseraceae *Boswellia serrata* **Sudan**	Frankincense	*Triterpenoid gum-resin* Pale yellow-brown. Small ‘beads’. Opaque. Strong scent.		Bristol Botanicals Ltd. Reference GAW009
STOBB	Altingiaceae *Liquidambar orientalis* **Turkey**	Storax	*Triterpenoid balsamic resin* Pale yellow/orange. Viscous liquid. Strong scent.		Bristol Botanicals Ltd. Reference GAW017
STOSLB	Altingiaceae *Liquidambar orientalis* Bark **Turkey**	Storax	*Resinous bark* Red-brown. Variable fragments. Moderate scent.		Soma Luna Reference *L. orientalis* bark
STOSLR	Altingiaceae *Liquidambar orientalis* Raw extract **Turkey**	Storax	*Triterpenoid balsamic resin* Dark yellow/brown. Viscous, slightly opaque. Strong scent.		Soma Luna Reference INC151

STOSLP	Altingiaceae *Liquidambar orientalis* Purified extract **Turkey**	Storax	*Triterpenoid balsamic resin* Yellow/orange. Viscous, clear. Strong scent.		Soma Luna Reference INC084
STO459	Altingiaceae *Liquidambar orientalis* **Not given**	Storax	*Triterpenoid balsamic resin* Brown/black. Amorphous. Opaque. No scent.		Royal Botanical Gardens, Kew Reference KEBC56459
STO105	Altingiaceae *Liquidambar orientalis* **Not given**	Storax	*Triterpenoid balsamic resin* Orange/brown. Amorphous. Opaque. No scent.		Royal Botanical Gardens, Kew Reference KEBC56105
STYBNT	Styraceae *Styrax officinalis* **Cornwall**	Styrax	*Extract of pulverised twigs.* Twigs from sapling. NB: No exudate visible when 'wounded' bark.		Burncoose Nurseries Product number 4243 Sapling grown in UK, purchased by author 2012

These comprised botanically and geographically certified samples purchased for this project (Bristol Botanicals Ltd.; Soma Luna LLC), materials which had been curated in reference collections for many years (Royal Botanical Gardens, Kew; Molecular Sciences Laboratory, University of Bradford) and freshly-collected *S. officinalis* twigs (author's tree, Burncoose Nurseries, Cornwall). The aim was to provide comparable chromatographic and mass spectral data to facilitate the identification of any resinous substances indicated by the presence of suites of phenolic and/or terpenic compounds in the archaeological samples. The naturally aged resins were selected to assist in the evaluation of degradative changes and in the authentication of poorly preserved archaeological materials. In combination with information compiled from the phytochemical and archaeological literature a database of diagnostic biomarkers detailing their molecular and key fragment ions was compiled for future reference (**Appendix 3**).

6.6 Synthetic chemistry

A series of triterpenoids in samples from the focal burial at the Eagle Hotel site, Winchester, UK (**7.5**) illuminated the need to distinguish between isomers/epimeric pairs. As only the 3β-epimers could be readily purchased and in order to acquire additional laboratory skills, the author received training from Dr William Martin in the synthesis of organic compounds and use of nuclear magnetic resonance (NMR) spectroscopy.

6.6.1 Synthetic method

Standards of 3β-oleanolic acid (CAS 508-02-1) and 3β-ursolic acid (CAS 77-52-1) were purchased from Tokyo Chemical Industries Ltd., UK. Approximately 20 mg of each was weighed out, dissolved in acetone and oxidised using Jones reagent ($Na_2Cr_2O_7$, H_2SO_4) to synthesise the corresponding ketones, oleanonic and ursonic acid). Progress of the reaction was monitored by thin layer chromatography (TLC). Once complete, the reaction was quenched with MeOH and the product extracted by partitioning between diethyl ether and water, dried over anhydrous sodium sulfate (Na_2SO_4), filtered and evaporated *in vacuo* (rotary evaporator and HiVac).

One portion was retained for GC-MS analysis and the remainder converted to the 3α-epimeric alcohol. This was achieved through the use of a novel approach (proposed by Dr Martin) with dissolution in tetrahydrofuran (THF) and diastereoselective reduction of the ketone using a bulky reducing agent, *L*-selectride® (CAS 38721-52-7; 1M $C_{12}H_{28}BLi$). Progress of the reaction was monitored by TLC and quenched with dilute citric acid. The product was extracted by partitioning between DCM and water, dried over anhydrous Na_2SO_4, filtered and evaporated *in vacuo.* Purification was carried out by flash column chromatography (petroleum ether: ethyl acetate, 8:1, v/v). Yields were ~95% and ~70% respectively (**Figure 6.4**).

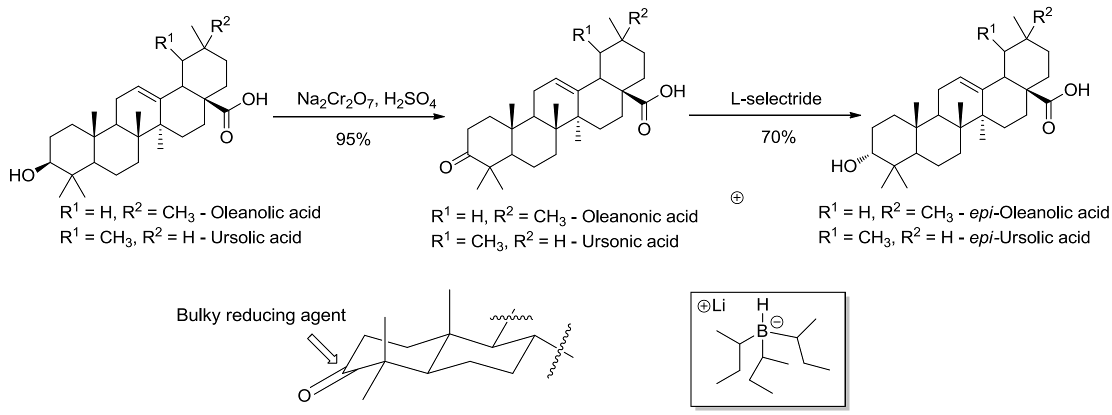


Figure 6.4. Scheme of the synthetic method employed (created by author with assistance of Dr W.H.C. Martin).

6.6.2 Nuclear magnetic resonance (NMR) spectroscopy

NMR spectroscopy provides "*information about the number of magnetically distinct atoms of the type being studied...and the nature of the immediate environment of each type*", with hydrogen and carbon most commonly assessed (Lampman *et al.* 2010: 105). Although traditionally used, as here, to establish the molecular structure of individual, purified compounds (e.g. Başar *et al.* 2003; Charrié-Duhaut *et al.* 2007; Hovaneissian *et al.* 2008; Mathe *et al.* 2004a; Nicholson *et al.* 2011), NMR has also been employed in the characterisation of complex materials. For example, it has been used to determine the botanical source of amber (fossilised resin) (Lambert *et al.* 1996; Langenheim 1996) and to distinguish between modern plant exudates (Lambert *et al.* 2005, 2007). Recently, these applications have been extended to include the identification of input sources to a modern unguent made to an ancient recipe (Colombini *et al.* 2011) and of archaeological resins (Bruni and Guglielmi 2014). Results indicate that NMR, in conjunction with FTIR and/or GC-MS, may have considerable potential for future research into the use of plant exudates in antiquity.

In order to establish structural information, an aliquot of each of the six standards was diluted in deuterated chloroform ($CDCl_3$). 1H NMR spectra were obtained on a multi-nuclear Bruker Avance spectrospin 400 ultrashield FT NMR Spectrometer fitted with a QNP 5mm variable temperature probe and BACS autosampler. Assignment of the multiplicity of overlapping resonances that appear upfield as a result of the complexity of these molecules was not undertaken. Instead, determinations were made through evaluation of the downfield region of the spectrum and focussed on the presence or absence of resonances (two multiplets between 2.3-2.7 ppm) arising from a deshielded ketone at C-3 on the A ring.

6.6.3 Atmospheric pressure chemical ionisation-liquid chromatography-mass spectrometry (APCI-LC-MS)

The opportunity to send the six standards to Dr Richard Gallagher at AstraZeneca, Alderley Park, Cheshire, UK for further analysis arose, thanks to Dr Martin and Dr Richard Bowen. APCI-LC-MS is an instrumental technique which nebulises and vaporises the sample in solution using heat and nitrogen gas. The resultant plasma is then ionised (positive or negative mode) by a coronal discharge electrode with the mobile phase acting as the chemical ionization reagent (Harris 2007: 490-491). This is a 'softer' method than EI so less fragmentation of the analyte occurs resulting in enhancement of the molecular ion and primary fragment ions (Gross 1986).

Sub-samples were dissolved in 50:50 (v/v) water:methanol (~0.01 mg/ml, 10 µl injection) and eluted along a gradient (T=0.0, A=95%, B=5%, T=9.0, A=50%, B=50%, T=9.01, A=2%, B=98%, T=11.0, A=5%, B=95%) using a mobile phase of 0.05% aqueous formic acid (A): 0.05% formic acid in methanol (B) with a flow rate of 0.45 ml/min. Analysis was carried out using a hybrid linear ion-trap (Orbitrap XL) mass spectrometer (ThermoScientific) connected to an Acquity ultra high performance liquid chromatography system fitted with an Acquity UPLC BEH C18, 1.7 μm, 2.1 mm x 100 mm column (Waters Ltd). Data was collected in atmospheric pressure chemical ionisation (APCI) positive ion mode (vaporiser 250 °C; capillary needle 6 kV; gas flow 50:5 auxiliary flow; source capillary 275 °C). The MS was set to alternate (~25 sec) between recording a mass spectrum over *m/z* 100 to 800 amu (7.5k FWHM resolution) and an higher energy collision dissociation product ion mass spectrum (7.5k FWHM; normalised collision energy 40eV) from the parent mass down to *m/z* 50 (Gallagher 2014, pers. comm., 19-24 March).

As a result of initial observations, further work was undertaken by Chloe Townley (supported by a British Mass Spectrometry Society (BMSS) summer studentship) and is reported in Townley *et al.* (2015; **Appendix 7.6**). These findings did not directly facilitate the identification of archaeological materials. Nonetheless, they resulted in discussions concerning the ease and mechanism by which epimerisation might occur as a result of interactions in the burial environment which may assist in this endeavour (**7.5**).

6.7 Summary

Samples were collected from target burials from Roman Britain that were extant, able to be accessed and which, on evaluation, contained materials suitable for organic residue analysis (**Table 4.2**). The samples collected included visible resinous fragments, amorphous residues adhering to skeletal elements and plaster body-casings and comminuted grave deposits from within the burial containers. In addition, where possible, control samples were selected to provide background values indicative of soil components and contaminants. The most appropriate solvent extraction, derivatisation and instrumental methods of analysis in relation to the requirements of the research agenda were ascertained through a review of the literature and a sample protocol was established (**Figure 6.1**). Reference materials and standards were purchased, obtained from reference collections or synthesised to facilitate the characterisation of any resinous substances present (**Table 6.4**; **Appendix 3**) and to address specific questions that arose as the project progressed (**7.5**). Identification was carried out in conjunction with previously published mass spectral data and contextualised in relation to the exudates available within the Roman Empire (**Chapter 3**), with only botanically identified and, where possible, geographically appropriate exudates selected. Analytical results are detailed in the following series of case studies (**Chapter 7**) and accompanying appendices (**Appendix 6.1-6.10**) with TICs available on **Disc 1**.

Chapter 7

Case studies: background and results

"they washed and anointed the cold body of their friend and lamented. When they had wept enough, they placed him on the bier and covered him with his own purple robes".
Virgil, Aeneid 6:218-220 nd

7.1 Introduction

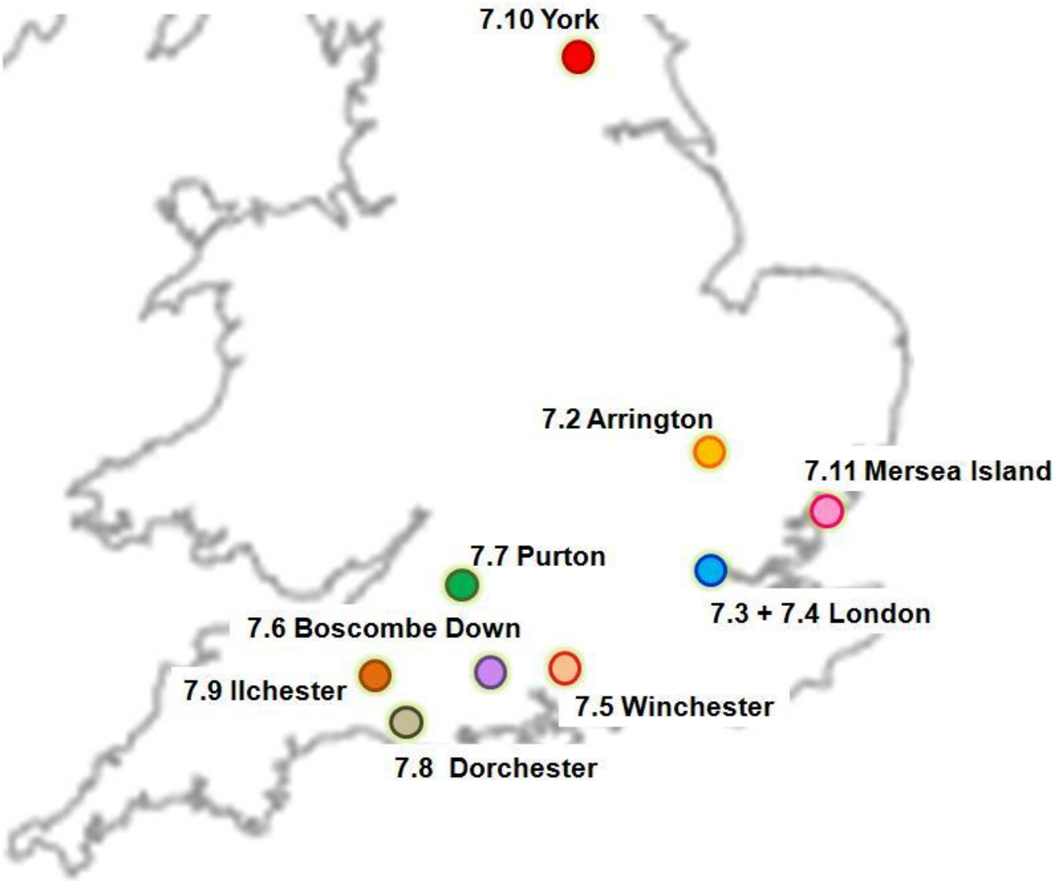


Figure 7.1. The locations across Britain from which samples were obtained for chemical analysis, the numbers relate to the relevant case studies, below (created by author).

Results of the chemical analysis of materials collected from the various Roman period mortuary contexts selected for this project are presented as a series of case studies. The geographical locations of the sites, from Britain, sampled are shown in **Figure 7.1**. General and site specific background information is provided in order to place each burial in context with details regarding the samples selected and the analytical findings discussed in the text. These are supplemented by key images, total ion current (TIC) and extracted ion current (XIC) chromatograms, mass spectra and tabulated data. Additional tables listing all the information collated and the results from each burial can be found in **Appendix 6** with TICs of every sample made available on **Disc 1.** Where terpenic compounds were found to be present, these were identified through comparison with the published literature and the analysis of modern reference materials (**Table 6.1**). Key results obtained from the latter are presented (TICs and XICs accompanied by tables of mass spectral data and structural information) by botanical source in **Appendix 3**.

7.2 Case Study 1: Infant, Arrington, Cambridgeshire (Brettell *et al.* 2014)

7.2.1 Background

In 1990, during the laying of a sewage pipe, an apparently isolated inhumation was discovered at Wraggs Farm, Arrington, Cambridgeshire, UK (TL 3269 5049). This Roman period burial was situated close to the line of Ermine Street, between the posting station at Arrington Bridge (2^{nd}-early 5^{th} century AD) and the small settlement at Wimpole (1^{st}-4^{th} century AD) (Taylor 1993). Interred in a lead-lined oak coffin and orientated with head to the west, the skeletal remains were found to be those of an approximately twelve month old infant (determined from the dentition and stage of fusion of the post-cranial elements). Originally reported to have been hydrocephalic, re-examination showed that cranial morphology did, in fact, fall within expected parameters for an infant of this age. Thus, the only pathological changes visible comprised bilateral *cribra orbitalia*, often indicative of iron-deficiency anaemia as a result of infection (Appleby 2011; **Figure 7.2**).

Although no grave goods were found within the coffin, a number of pipe-clay figurines associated with a 'brown peaty' mass, probably a wooden box, rested on the coffin lid at its foot. These mould-made anthropomorphic and animal figures were mass produced in factories in Gaul and the Rhineland and probably date to the 2^{nd}-3^{rd} centuries AD on stylistic grounds (Bémont *et al.* 1993: 131; Green 1993). Most have been found in association with graves but they also appear to have been used in household shrines and were deposited in sacred springs and sanctuaries as votive offerings or to mark a rite of passage (Rouvier-Jeanlin 1972: 27-29). Accordingly, their presence with the Arrington infant almost certainly had ritual significance.

Figure 7.2. Skeletal remains of the Roman period infant (MAA 1994.19) from the lead-lined coffin with pipe clay figurines, Arrington, Cambridgeshire: a. cranium; b. maxilla; c. mandible; d. frontal showing *cribra orbitalia* in orbits (Author's images). Scale bars: 1 cm.

In addition, exceptional preservation conditions within the lead-lined coffin had resulted in the survival of a small dense mass of matted human hair at the apex of the cranium together with fragments of dyed wool, which appeared to have been folded over and held together by a white substance. A white substance, variously reported as lime or lead carbonate, also formed a layer on the base of the coffin while the cranial vault was filled with sticky greyish clay. Excavation of this mass of material within a laboratory setting revealed the presence of *"numerous pieces of aromatic resin, still smelling distinctly of incense*" (Taylor 1993: 194). Initial analysis indicated that these fragments were, indeed, representative of the inclusion of a resin within this burial but further identification was not undertaken (Taylor 1993).

7.2.2 Sample selection

The resinous materials (Acc. No. 1994.21; **Figure 7.3**) from the infant burial (1994.19) at Arrington were accessed with permission of the Museum of

Anthropology and Archaeology (MAA), Cambridge, UK facilitated by Imogen Gunn. Ensuring that fragments were retained for future analysis, a representative portion (four pieces) was selected for analysis by FT Raman spectroscopy and GC-MS. The extract was partitioned with one portion silylated and the other methylated to facilitate comparison with the published literature. Additional details can be found in **Appendix 6.1**, TICs of the silylated and methylated extracts on **Disc 1, File 6.1** and comparative data from modern reference resins in **Appendix 3.2.**

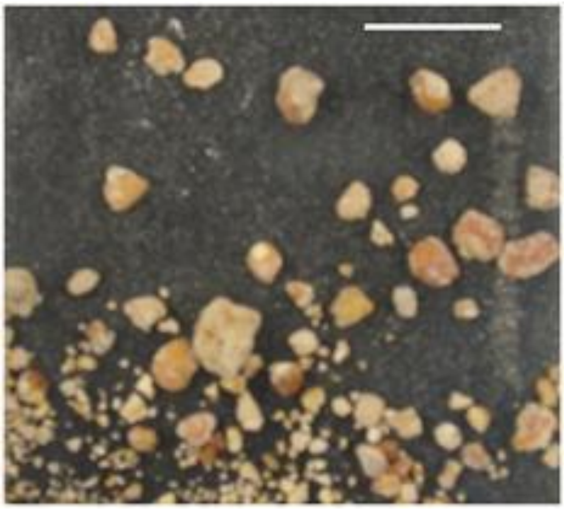


Figure 7.3. The resinous fragments recovered from the Roman period lead-lined infant burial from Arrington, Cambridgeshire (Author's image). Scale bar: 1 cm.

7.2.3 Results

7.2.3.1 Sample description

The four fragments selected ranged from yellow-orange to orange-red in colour and were variable in size. No scent could be detected. They were also brittle and highly friable so that even careful handling with tweezers readily reduced them to powder. They dissolved fully in the organic solvent to produce a yellow-orange solution.

7.2.3.2. Initial findings: FT Raman spectroscopy

Initial analysis using FT Raman spectroscopy provided a spectrum with a limited number of features (**Figure 7.4**). This is consistent with the aged nature of the sample. The bands observed denote the presence of various hydrocarbon and carbon-carbon bonds. The most intense, a broad strong peak at c. 2930 cm^{-1} is representative of $\nu(CH_2)$ and $\nu(CH_3)$ stretching modes while the spectral feature at c. 1440-5 cm^{-1} is assigned to $\delta(CH_2)$ and $\delta(CH_3)$ deformations with $\nu(CH_2)$ scissoring. The medium peak with shoulder at 1649 cm^{-1} shows the presence of conjugated $\nu(C=C)$ bonds characteristic of terpenic acids. These features are common to most resins and gum-resins although their shape and intensity varies in relation to the degradation state of the sample (Brody *et al.* 2002). The spike at c. 1495 cm^{-1} is an artefact of the instrument.

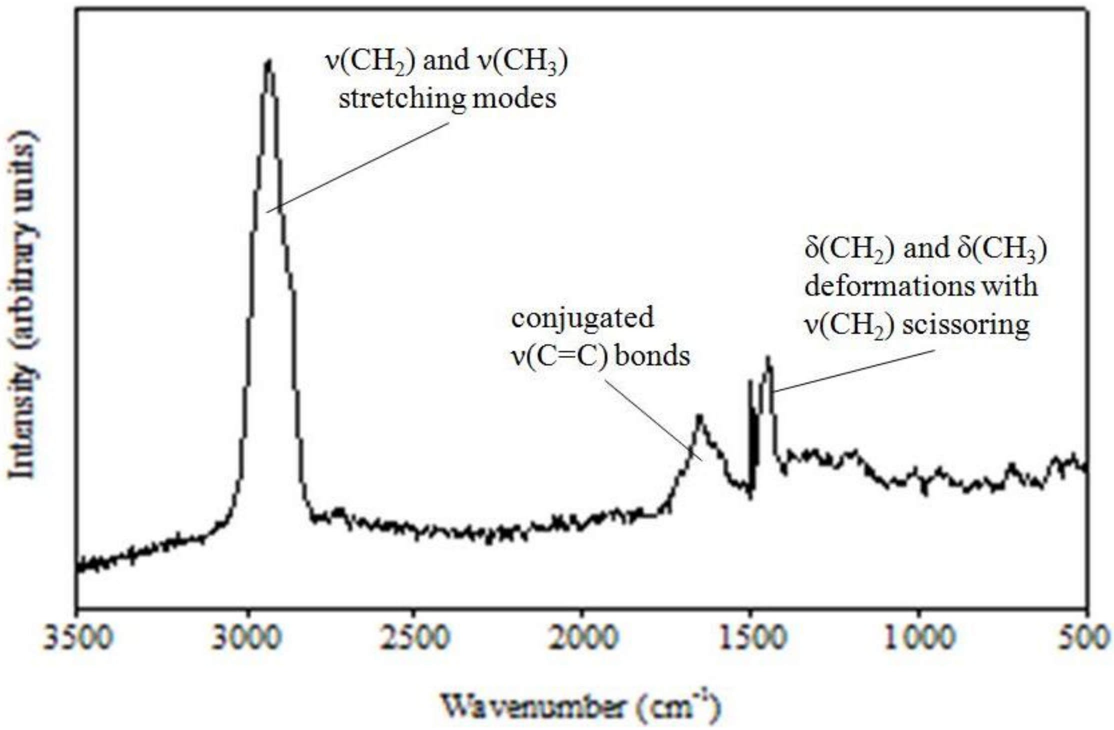


Figure 7.4. FT Raman spectrum of the resin found with the Arrington infant, Cambridgeshire wavenumber region 3500-500 cm^{-1}, $Nd^{3+/}$YAG laser excitation at 1064 nm, nominal laser power 270 mW, 4 cm^{-1} spectral resolution, 1000 scans accumulation.

7.2.3.3 Characterisation: GC-MS

GC-MS analysis proved considerably more effective in the characterisation of this resin and showed that the sample contained a range of triterpenic compounds (**Figure 7.5**). Methylation confirmed that these moieties had oleanane and tirucallane skeletons based on their characteristic fragmentation patterns resulting from an initial *retro*-Diels-Alder (*r*DA) reaction in the C ring and subsequent rearrangements, as detailed in Assimopoulou and Papageorgiou (2005a; **Figure 7.6; Table 7.1**). Peaks 6, 7, 9 and 10 were identified as moronic (MA), oleanonic (ONA), *iso*masticadienonic and (IMDA) masticadienonic acid (MDA), respectively. These are biomarkers for resins from the genus *Pistacia* (**5.3.3; Table 5.4).**

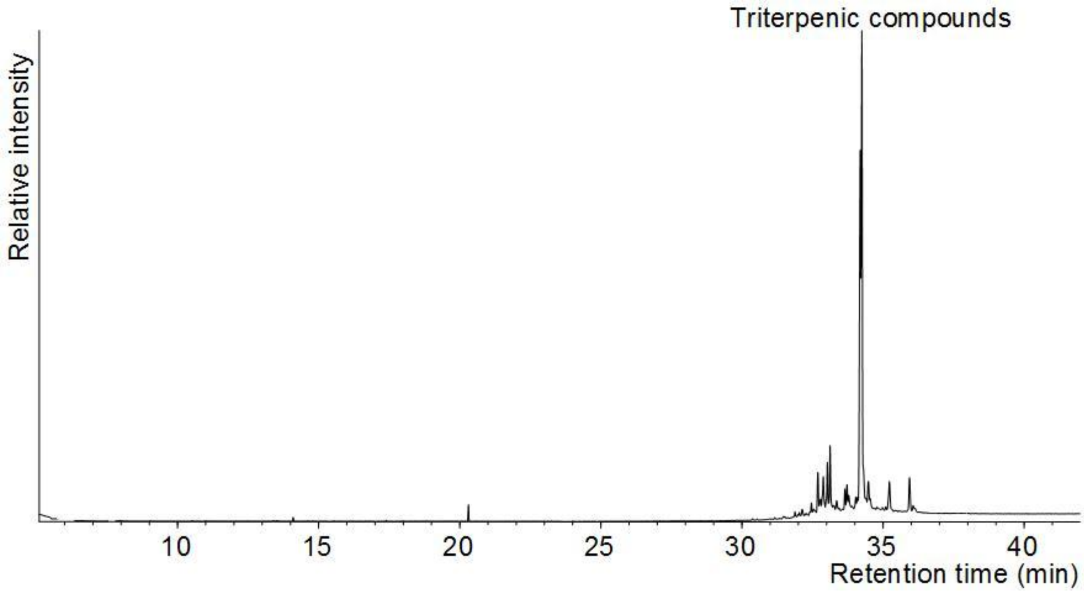


Figure 7.5. TIC resin fragments, infant inhumation, lead-lined coffin, Arrington Cambridgeshire, UK showing the dominance of triterpenic compounds (TMS derivatives).

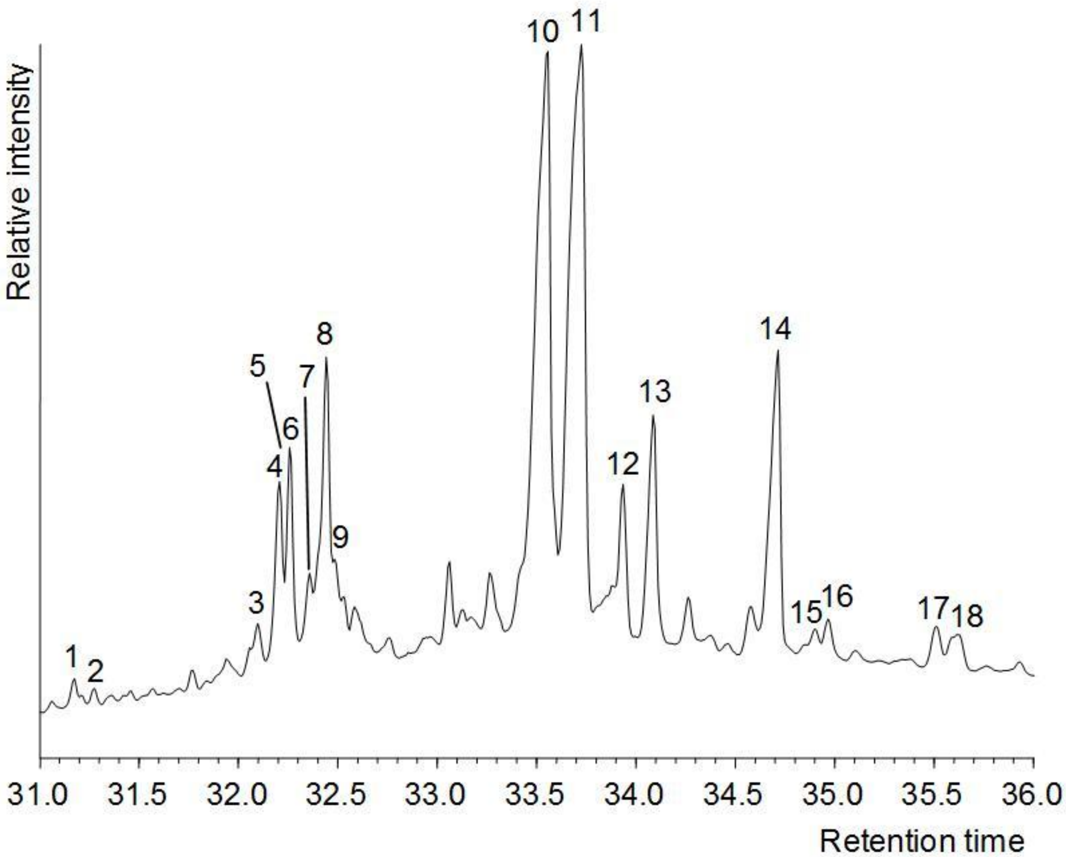


Figure 7.6. Partial TIC (31-36 min) triterpenic compounds (methyl esters), resin fragments, infant inhumation, lead-lined coffin, Arrington Cambridgeshire, UK. Peak numbers relate to **Table 7.1**.

Table 7.1. Identification of the triterpenic compounds (methyl esters), resin fragments, lead-lined coffin, Arrington, Cambridgeshire based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
1	442	204	431, 355, 281, 253, 189, 175, 135, 81	3,4-seco-28-norolean-12(18)-en-3-oic acid
2	442	163	427, 405 ,355, 281, 229, 207, 191, 119	3β-hydroxy-6β-hydroxymethyl-28-norolean-17-ene
3	426	411	393, 327, 281, 241, 207, 189, 135, 121	tirucallol
4	410	204	393, 369, 313, 281, 253, 189, 175, 133	28-norolean-12-en-3-one
5	424	218	413, 355, 327, 281, 207, 203, 189, 147	β-amyrenone
6	440	189	413, 281, 262, 251, 207, 203, 163, 133	olean-18-enolic aldehyde (morolic aldehyde)
7	408	408	393, 377, 281, 259, 241, 202, 189, 173	28-norolean-12,17-dien-3-one
8	440	203	413, 262, 251, 218, 189, 175, 133, 119	olean-12-enolic aldehyde (moronic aldehyde)
9	410	163	395, 281, 253, 218, 207, 191, 175, 133	28-norolean-17-en-3-one
10	468	189	453, 409, 391, 262, 249, 203, 163, 119	moronic acid
11	468	203	453, 409, 282, 262, 249, 189, 133, 119	oleanonic acid
12	438	203	409, 257, 232, 189, 175, 133, 119, 105	oleanonic aldehyde
13	468	453	421, 355, 313, 257, 245, 231, 161, 121	*iso*masticadienonic acid
14	468	453	393, 355, 313, 257, 245, 231, 161, 127	masticadienonic acid
15	512	437	497, 355, 341, 315, 241, 301, 189, 121	3α-acetoxy-3-epi*iso*masticadienolic acid
16	470	455	437, 341, 301, 241, 229, 161, 127, 95	3-epi-masticadienolic acid
17	482	217	467, 454, 385, 317, 276, 257, 189, 119	oxidised oleanonic acid derivative
18	512	437	497, 409, 355, 315, 241, 229, 189, 127	3α-acetoxy-3-epimasticadienolic acid

Further evaluation was made through comparison with a number of modern *Pistacia* resins with the focus on the two main resin-producing species, *P. lentiscus* (Greece) and *P. terebinthus* (Iran) (**Appendix 3.2**). A clear correspondence between the mass spectra of the biomarkers in the modern resins and the archaeological sample confirmed the latter to be a *Pistacia* spp. resin. The four main resin acids (MA, ONA, IMDA and MDA) and their derivatives are present in exudates of all members of the genus *Pistacia* which makes lower taxonomic determination problematic. Some minor components may differ, however, and provide indicators as to species, with the proviso that the natural variability dictates caution, especially when dealing with archaeological materials (Assimopoulou and Papageorgiou 2005a, 2005b). Thus, the presence of 3,4-seco-28-norolean-12(18)-en-3-oic acid (1) and olean-18-enolic aldehyde (3) in the Arrington fragments suggests that the source is more likely to be *P. terebinthus* (or a closely related species) than *P. lentiscus.*

Comparison with the reference materials also permitted the state of preservation and likelihood of anthropogenic pre-treatment prior to deposition to be assessed. Both the modern *P. lentiscus* sample and the Arrington resin showed a slight predominance of oleanonic acid, the absence of ocotillone-type molecules (oxidised dammaranes with a base peak at *m/z* 143 which are prevalent in aged *Pistacia* spp. resins; Stern *et al.* 2003) and the presence of compounds (e.g. peaks 2, 3, 15 and 18) that are generally absent from archaeological materials due to natural degradation processes. In contrast, the modern *P. terebinthus* sample was both visually (dull, opaque) and chemically degraded as demonstrated by the poorly resolved ‘hump’ in its chromatogram, increase in moronic acid, low abundance of other resin acids (e.g. masticadienonic and *iso*masticadienonic acid), presence of ocotillone-type molecules and absence of many of the compounds recorded in fresh resins (cf. Assimopoulou and Papageorgiou 2005b). This combination, symptomatic of naturally aged materials, highlights the unusual level of preservation observed in the archaeological sample and permits a high degree of confidence in the botanical determination.

It also indicates a lack of pre-treatment in antiquity. There is, for example, no evidence of the resin fragments having formed part of an admixture. In addition, although their condition can, in part, be attributed to the protection afforded by the lead-liner the resin does not appear to have been heated. Research has shown that, in *Pistacia* spp. resins, an abundance of 28-norolean-17-en-3-one and/or the presence of a series of unidentified compounds with a characteristic mass fragment at *m/z* 453 (in methylated samples) provide evidence of thermal alteration (Serpico and White 2000; Stern *et al.* 2003). In this instance, the functionalised resin acids had remained dominant while these proposed markers of heating were either in low abundance (5) or absent (compounds with base peaks at *m/z* 453) as was the case with the untreated modern reference resins analysed.

7.2.4 Summary and interpretation of findings

Chemical analysis of amorphous masses found around the cranium of a year old infant interred a lead-lined coffin near Arrington, Cambridgeshire conclusively demonstrated that these comprised *Pistacia* spp. resin fragments. Some minor components indicated that this exudate may have derived from *P. terebinthus* (or a closely related species), rather than *P. lentiscus*. The range of compounds present suggested that this aromatic exudate had been deposited in its natural state as it was exceptionally well preserved, unadulterated and showed no evidence of thermal alteration.

7.3 Case Study 2: The cemeteries of Roman London (Brettell *et al.* 2015a)

7.3.1 General background

London (*Londinium*), Middlesex, UK is thought to have been founded by the Romans around AD 50. Considerable evidence for occupation exists from the Claudian period in the form of coins and imported pottery (Barber and Bowsher 2000: 299; Marsden 1980: 9-13). This new settlement was a key part of the developing road system and appears to have been designed as a trading centre with quays along the river frontage and bridges established across the Thames (Mattingly 2006: 256-259, 273-274; Merrifield 1969: 22-67). Its importance, even at this early period, is attested by the fact that the town was one of the targets of the Boudiccan Revolt (AD 61) and was subsequently rebuilt, becoming the largest urban site in *Britannia*. Indeed, the prime location of *Londinium* seems to have led to its development as the administrative centre of the province. Archaeological excavations have provided considerable evidence of an abundance of imported goods (emphasising its role as an emporium), manufacturing activity and for the construction of substantial public buildings. Thus, during the late 1st-early 2nd centuries AD, this *municipium* (or perhaps honorary *colonia*) acquired a basilica and forum (subsequently rebuilt on a massive scale), bathhouses, an amphitheatre and a stone fort at Cripplegate (Mattingly 2006: 265-276; Merrifield 1969: 68-82; Potter and Johns 1992: 79-80, 106).

Although some areas then declined, others flourished and the town continued to prosper becoming the capital of *Britannia Superior* and seat of the governor and/or his officials after the division of the province in AD 197. This period also saw the construction of town houses with hypocausts, painted wall plaster and mosaics and the restoration and building of a number of temples (Marsden 1980: 49-130; Mattingly 2006: 275-276, 281-282). A defensive wall was then added in the 3rd century AD (with later modifications) and a mint operated in the city from c. AD 288-326 (Merrifield 1969: 84). This prosperous urban settlement, in common with others in Britain, underwent a significant period of change after AD 350 and effectively ceased to function soon after AD 400 with the last Roman period burials dated around the turn of the century (Barber and Bowsher 2000: 304-307; Potter and Johns 1992: 211-212).

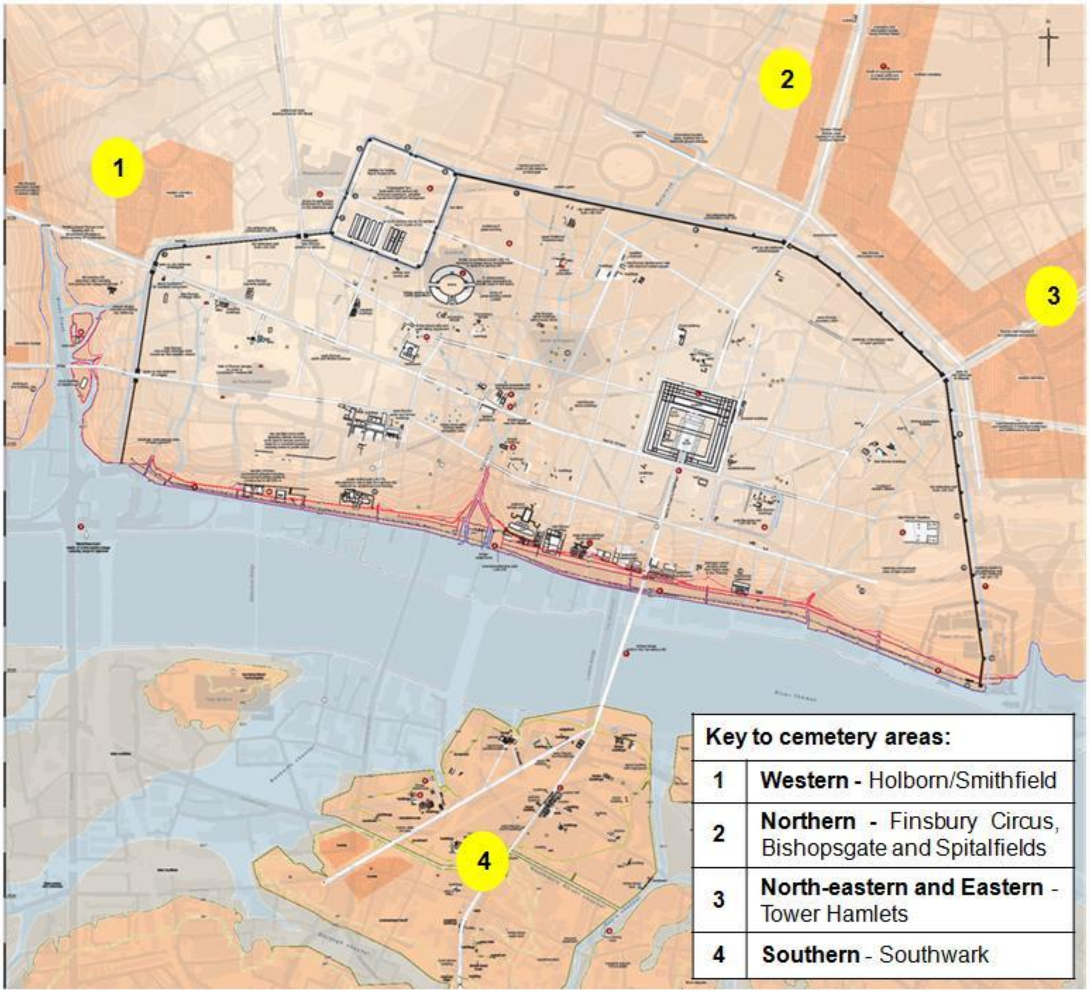


Figure 7.7. Roman *Londinium* superimposed on modern London, showing the location of the main Roman burial areas outside its walls and in its suburb of Southwark (MoL 2014).

Following Roman law, the disposal of the dead took place in burial grounds located outside the walls of the city (**Figure 7.7**). Situated along the roads leading to and from *Londinium*, the main burial area groupings (represented by c. 190 sites containing over 200 cremations and 1100 inhumations) have been discussed by Hall (1996) with more recent discoveries reported in the Museum of London monograph series (cited below). These areas comprise:

- the western burial grounds which extended between the Roman gates at Newgate and Aldersgate and were focused upon the region that is now Holborn and Smithfield (Bentley and Pritchard 1982; Watson 2003);

- the northern burial ground which spread around Finsbury Circus, Liverpool Street Station and Spitalfields Market, roughly along the line of Bishopsgate (Evans and Pierpoint 1986; Swift 2000; Thomas *et al.* 2003);
- the north eastern burial ground situated along the main road to Colchester (Hall 1996) and the eastern burial ground stretching from the area of the Minories possibly as far as (Brod)Love Lane off Cable Street (Barber *et al.* 1990; Barber and Bowsher 2000; Ellis 1985; Whytehead 1986);
- the southern burial grounds distributed along the roads to/from the suburb of Southwark (Cowan 2003; Dean 1981; Dean and Hammerson 1980; Mackinder 2000).

Funerary inscriptions show that the people of *Londinium* and its hinterland interred in these burial grounds originated all over the Empire and included legionary soldiers, the wives of provincial slaves and even a procurator, *Julius Classicianus*, who is also known from literary sources (Barber and Bowsher 2000: 312-313; Mattingly 2006: 296-301). Discovered over many centuries, older finds frequently suffer from inadequate recording with skeletal remains reburied and accompanying artefacts and containers either lost or sold (Hall 1996). Moreover, continual occupation has resulted in considerable disturbance and destruction of archaeological evidence (Evans and Pierpoint 1986). Nonetheless, what survives indicates that a variety of burial practices were employed, as elsewhere in the Empire, with cremation dominant during the 1st-3rd centuries AD and an increase in inhumation beginning in the 2nd century AD (Merrifield 1969: 180-181). By the later Roman period, the majority were interred supine, extended, in wooden coffins with few, if any, surviving grave goods (Barber and Bowsher 2000: 84-95; Watson 2003: 7-8). Examples of decapitations, multiple and prone burials have also been identified alongside a variety of packing materials (stone, tile, chalk) and burial containers (tile cists, stone sarcophagi, lead-lined coffins) with some indications for the presence of mausolea (Barber and Bowsher 2000: 101-104, 300-301; Cowan 2003: 73; Hall 1996; Mackinder 2000: 19-23; Thomas *et al.* 2003).

Thus, within each burial area certain individuals were accorded a more elaborate mortuary treatment than the 'norm' (**Table 7.2**). These material manifestations of difference, generally dated to the late 2^{nd}-4^{th} centuries AD, comprised inhumation in stone sarcophagi and/or lead-lined coffins in accordance with the class of burial proposed by Ramm (1971). In addition, a considerable number of plaster burials have been identified many of which were associated with interments in wooden coffins (Barber and Bowsher 2000: 101-104). As these elements may indicate an attempt at body preservation (Sparey Green 1977), samples were sought from extant remains. Soil samples from normative inhumations from West Smithfield in the western burial ground (Bentley and Prichard 1982) were also analysed to provide a comparative sample set (reported in Harrison 2014, *et al.* 2013).

Table 7.2. Roman period inhumations in stone sarcophagi and/or lead-lined coffins and/or with plaster from each London burial area (compiled from Barber and Bowsher 2000; Cowan 2003; Hall 1996; Mackinder 2000; Museum of London 1999; Whytehead 1986).

Burial area	Stone	Lead	White material
Western	4	3	2
Northern	8	4	5
North east/eastern	3	6	89
Southern	0	1	19

7.3.2 Sample selection

The skeletal remains from thirty-two graves were evaluated (**Table 7.3**). Thirty-six samples were selected from twenty-two of these burials (not including the 'Spitalfields Lady', Case Study **7.4**) with the kind permission of the Museum of London (MoL), facilitated by Dr Rebecca Redfern. These materials consisted of residues adhering to skeletal elements and debris (grave deposits) loose within the plastic storage bags. Visual assessment suggested that many of these materials were predominantly inorganic in nature (e.g. white residues) while others comprised adhering soil. Nonetheless, to be certain, representative portions were selected for analysis. The bulk of these results are summarised below, **7.3.3.1**, with the data from each burial tabulated and discussed by cemetery grouping and site, accompanied by additional background information and images, in **Appendix 6.2.** Findings of potential archaeological relevance are presented in **7.3.3.2.** TICs can be found in **Disc 1, File 6.2**.

Table 7.3. Details of samples collected from the cemeteries around London (for images, TICs and tabulated results see **Appendix 6.2**).

Site location	Site information	Burial	Osteological details	Burial details	Sample information
Burials to the west of London					
ATC97; TQ 3153 8159; Watson 2003					
Atlantic House Holborn Viaduct, London, EC1	*Located:* along the road to Silchester beside the River Fleet *Recovered:* 29 cremation and 19 inhumation burials *Observations:* disturbed; most in wooden coffins	Context 216 Burial 4 in Watson 2003	Elderly male EW orientated Near edentulous mandible Very fragmented	AD 140-200 Ceramic vessel Chicken bones Charcoal ?intrusive	Sample representative of dark staining reported from around the burial not found ATC216M from mandibular foramen ATC216L from leg bone fragments
		Context 261 Burial 7 in Watson 2003	Male, 18-25 years Good preservation Most bones present Minimal breakage	AD 140-250 No grave goods Ceramic sherds and cattle horn core in grave fill	Black residues reported; distributed on skeletal elements; tar-like substance, desiccated outer surface, glossy/sticky inner surface ATC261F residue from distal right femur ATC261C residue from left distal clavicle ATC261A residue from left acetabulum
St Martin-in-the-Fields; TQ 3009 8053; Colman Getty press release 2006					
St Martin-in-the-Fields City of Westminster, London, WC2	*Located:* burial ground to the west of Roman London *Recovered:* 20+ Roman and many medieval burials *Observations:* extensive 19th c. AD disturbance	Sarcophagus burial damaged by Victorian sewer	Body disturbed Head removed	Late 4th-early 5th c. AD Stone sarcophagus	Skeleton not accessioned. Grave deposits washed in IMS. Samples from base of sarcophagus: 1. head end 2. middle portion 3. foot end
The northern burial ground					
SRP98; TQ 3348 8189; Thomas 1999; Thomas *et al.* 2003				***NB: discussed in 7.4***	
'Spitalfields Lady' 280 Bishopsgate + the Spitalfields Ramp, London, E1	*Located:* east of Ermine Street; on a slight rise *Recovered:* 2 cremations; c. 60 grave cuts, 53 with skeletal remains; 4 stone sarcophagi (3 robbed/damaged); plaster burial in timber-lined mausoleum	Site K Area 10 SK15903	Female, early 20s Well preserved Complete skeletal remains Supine, extended No pathologies Minimal dental wear	Mid 4th c. AD Barnack stone (limestone) sarcophagus from East Midlands Decorated lead (British) inner coffin Textiles (wool, silk); gold thread; bay leaves; items of jet and glass	21 spatially distributed samples collected: 7 flot (F), lightest fraction, from between the lead coffin and the sarcophagus 5 intended for particle size analysis (PSA), heavy fraction, between lead coffin and sarcophagus 9 intended for pollen analysis (P), grave deposits, within lead coffin, associated with the skeleton
BGB98; TQ 3335 8197; Swift 2000					
201 Bishopsgate London, EC2	*Located:* beside Ermine Street east of the Walbrook *Recovered:* six inhumation burials; both sexes; range of ages and grave goods *Observations:* mid 2nd-4th c. AD; some disturbance	B377	Mature adult male Extended, supine NS orientated Healed rib fracture; eburnation of ankle joint; Schmorl's nodes	On bed of crushed chalk	Bones unwashed, covered in yellowy-brown material, also filling cranium BGB377P, from pelvic bones BGB377S, associated with cranium
		B400	Child, 7-8 years Extended, supine NS orientated Moderate-poor preservation	Late 3rd-4th c. AD On a bed of crushed chalk Three copper alloy bracelets Fine wire near ankle	Bones unwashed, covered in concretions Yellow-brown with white fragments BGB400L, from leg bones BGB400C, associated with cranium

CDV99; TQ 3337 8143; Sankey and Connell 2008					
Premier Place Devonshire Square Houndsditch, EC2	*Located:* junction of Cutler Street and Devonshire Square *Recovered:* 35 inhumation burials (one double) *Observations:* majority extended, supine, WE orientated in wooden coffins; poorly preserved; disturbed	Context 146	Mature adult female Extended, supine WE orientated Fractured radius/metacarpal	Wooden coffin (nails) Plaster burial Fragments of ceramics in fill Sherd in mouth - residual	Yellow-brown residues on skeletal remains CDV146a, from right temporal and maxilla CDV146b, from left distal humerus
		Context 147	Mature adult female Lytic lesions in hands/feet; left ankle ankylosed (probably due to an erosive arthropathy)	No evidence of container No plaster or grave goods	Yellow-brown residues with orange flecks on skeletal remains CDV147, from os coxae and femora **NB: selected as comparative control**
Burial grounds to the east and north-east					
SCS83; TQ 3372 8106; Barber and Bowsher 2000; Barber *et al.* 1990; Ellis 1985					
9 St Clare Street London, EC3	*Located:* east of Roman city wall; site of Holy Trinity church, within the precinct of medieval abbey of St Clare *Recovered:* 4 inhumations; 1 with plaster packing; 1 with a ragstone surround; 2^{nd} c. AD cremation; flagons, ceramics and wooden box in a pit *Observations:* within precinct of medieval abbey; 1^{st}-4^{th} c. AD; ?mortuary structure	Site I, Grave 1 Skeleton 130	Adult Truncated, little remaining NS aligned Overlying grave 2	3^{rd} or 4^{th} c. AD Wooden coffin Plaster packing No grave goods	SCS130 material from bag and concretions from metacarpals and phalanges
		Site I, Grave 2 Skeleton 146	Adult male, 35-45 years WE aligned Partly overlying grave 3	Late 2^{nd} c. AD Wooden coffin Glass vessel in coffin Cooking pot beside coffin	SCS146 material from bags containing post-cranial elements
TTL85; TQ 3365 8140; Barber and Bowsher 2000; site record					
Three Lords Public House, 27 Minories, London, EC3	*Located:* east of the city wall; within the precinct of medieval abbey of St Clare *Recovered:* 3 inhumations; horse bones; ceramics *Observations:* remains of possible mortuary structure demolished late 3^{rd} c. AD	Site J, Grave 7 Skeleton 9	Subadult NS aligned Truncated, little remaining	Late 3^{rd}-4^{th} c. AD Wooden coffin (nails) Plaster coating Ceramics in fill	TTL009 debris from bags containing bones NB: skeletal elements cleaned
MSL87; TQ 3380 8110; Barber and Bowsher 2000					
49-59 Mansell Street London, E1	*Located:* east of the city wall; north of the proposed road through the cemetery *Recovered:* 61 cremations burials; 294 inhumations; animal burials; grave goods *Observations:* organised in rows; NS or EW aligned; some possible clusters	Site F, Plot 2 B355 (sk 720)	Robust male, 16-25 years Prominent muscle attachments on upper body	AD 180-400 Bead/scallop shell (pectin) decorated lead coffin Layer of plaster inside coffin	MSL720S, Dark patches from right scapula and radius NB: bones largely clean
		Site F, Plot 2 B392 (sk 754)	Sub-adult, c. 4-8 years Stunted growth; bowing of tibiae and fibulae, ?rickets; enamel hypoplasia and Harris lines	AD 250-350 Bead and reel decorated lead coffin High quality, grave goods: glassware; ivory figurine; Venus figurines; gold earrings; pyxis	Not found

PRE89; TQ 3400 8100; Barber and Bowsher 2000					
53-66 Prescot Street, Aldgate, London, E1	*Located:* east of the city wall; south of ?cemetery road *Recovered:* cremated bone; disarticulated remains; 4 inhumations; animal bone *Observations:* disturbance	Site H, Plot 18 B819, Context 20	?female, 26-45 years	AD 50-160 Chalk packing Chicken bones	Fragment of mandible only extant No residue
ETN88; TQ 3399 8102; Barber and Bowsher 2000; Barber *et al.* 1990					
East Tenter Street, London, E1	*Located:* east of the city wall; north of ?cemetery road *Recovered:* 2nd c. AD cremation burial; 8 inhumations; 3 with plaster; 3rd century AD grave goods *Observations:* fragments of a possible mausoleum	Site A, Grave cut 22 Context/sk 23	Adult (severely truncated) Extended, supine WE aligned	Wooden coffin (nails + stain) Plaster coating around remains	Too clean for sufficient sample to be obtained
		Site A, Grave cut 39 Context/sk 46	Adult Extended, supine WE aligned	Wooden coffin (timber stain) Plaster packing Green staining	ETN046a, material in nasal cavity ETN046b, material from bags containing post-cranial elements
		Site A, Grave cut 27 Context/sk 53	Adult Extended, supine WE aligned	Wooden coffin (timber stain) Plaster packing 2 jet pins; 1 bone pin	Too clean for sufficient sample to be obtained
AR72; TQ 3679 8360; Owen *et al.* 1973					
Armagh Road, Tower Hamlets, London, E3	*Located:* c. 125 m north of Roman Road (to Colchester); west of the River Lea *Recovered:* remains of 4 individuals, 3 in stone sarcophagi (one double burial/reused); 1 in a wooden coffin *Observations:* lids damaged by mechanical excavator but contents undisturbed; no other Roman period features recorded	Context 1 Skeleton I	Female, 36-45 years Extended supine WE aligned	3rd-4th century AD Limestone sarcophagus Calcium carbonate below remains Iron key; bone pin; jet pin; sherd	AR72/1a debris/grey-brown aggregations associated with cranium AR72/1b debris/grey-brown aggregations associated with post-cranial elements
		Context 2 Skeleton IV	Female, 25-35, truncated Extended, supine	Wooden coffin (nails)	Insufficient material present
		Context 3 [Marked up as 2] Skeleton III	Adult male Extended, supine NS aligned Well-healed fracture, left leg	Limestone sarcophagus ?secondary burial Incised lead attached to lid Calcium carbonate present	AR72/3 grey-white residues adhering to post-cranial elements
		Context 4 Skeleton II	Adult Partially disarticulated at base of limestone sarcophagus	Limestone sarcophagus ?original burial Incised lead attached to lid Calcium carbonate present	Insufficient material present
Burial areas to the south – the suburb of Southwark					
COSE84; TQ 3232 8039; Cowan 2003; Sidell *et al.* 2002					
Courage Brewery Park Street, SE1	*Located:* northern Southwark near the Bankside Channel *Recovered:* 7 inhumations; 3 reported as plaster burials *Observations:* probably in wooden coffins; disturbance and truncation	Site C , Burial 3 Context 1834	Adult female Relatively complete	Late 4th c. AD Wooden coffin, plaster burial	Skeletal remains scrubbed clean
		Site C, Burial 6 Context 1355	Robust adult male, 45+ years Periostitis on tibiae; osteophytes and Schmorl's nodes on vertebrae (?osteoarthritis)	Late 4th c. AD Wooden coffin, plaster burial Coin of Constantinopolis (c. AD 340) Ceramics (post AD 270)	Skeletal remains scrubbed clean
		Site C, Burial 7 Context 1503	Male, 35+ years (truncated) Healed fracture of clavicle	Late 4th c. AD Wooden coffin, chalk nodule	Skeletal remains scrubbed clean

GDV96; TQ 3275 7935; Mackinder 2000					
165 Great Dover Street Southwark, SE1	*Located*: c. 1km south of the River Thames, beside Watling Street *Recovered:* 1 bustum with food remains, ceramic lamps and incense burners; 4 other cremations and 25 inhumations *Observations*; considerable disturbance; 2 prone burials; 1 decapitation; all ages; both sexes; many without evidence of coffins; range of grave goods	Burial 17 SK126	Sub-adult c. 15 years Spina bifida occulta	Mid 2^{nd} c. AD Wooden coffin, bed of plaster Nene valley flagon (AD 150-250)	White residues SK126T, from ribs and vertebrae
		Burial 25 SK178	Robust adult male, 17-25 years Thickened cranial bones; navicular/1st cuneiform fused	Late 2^{nd}-3^{rd} c. AD Wooden coffin filled with plaster Glass disc (late 2^{nd}-3^{rd} c.AD)	White residues GDV178C, from cranium GDV178F , from foot bones
		Burial 26 SK150	Subadult c. 10-11 years	Late 2^{nd}-3^{rd} c. AD Wooden coffin filled with plaster Glass and jet items; hobnails	White residues GDV150R, from rib bones GDV150V, from vertebrae
		Burial 27 SK242	Adult female, 26-45 years Very heavy wear on dentition; osteophytes on thoracic vertebrae	Late 2^{nd}-3^{rd} c. AD Wooden coffin, filled with plaster No grave goods Finds of late 2^{nd}-3^{rd} c. AD in backfill	White and yellow residues GDV242P, from right parietal (white) GDV242L, from left tibia/fibula (yellow) GDV242C, from left clavicle (yellow)
LTU03; TQ 3225 7970; Ridgeway *et al.* 2013					
52-56 Lant Street, Southwark, SE1	*Located*: near the banks of an EW watercourse, to the west of Stane Street *Recovered:* 2 cremation burials; 84 inhumations; 9 with plaster *Observations:* most in wooden coffins; range of grave goods including high quality imported items	Burial no. BL43 Skeleton L292	Middle adult female Supine, extended Residual rickets (arm bones)	Wooden coffin (nails) Plaster in four areas around skeleton Copper-alloy bracelet; a hairpin	LTU292 debris from within plastic bags/residues from post-cranial elements
		Burial no. BL39 Skeleton L334	Young adult, female Supine, extended	Late 3^{rd}-4^{th} c. AD Wooden coffin (nails) Layer of plaster around skeleton Decorated Nene Valley beakers (x3); copper-alloy plate fragment	LTU334 debris from within plastic bags/residues from post-cranial elements
		Burial no. BL15 Skeleton L385	Adolescent (c. 14 yrs) Supine, extended Severe enamel hypoplasia; sleeve effect on molar; talon cusp on incisor; ?rickets	Late 2^{nd}-3^{rd} c. AD Wooden coffin, layer of plaster Ivory-handled knife; copper-alloy chain and key; bone casket with copper-alloy fittings; glass vessels	LTU334 debris from within plastic bags/residues from post-cranial elements
		Burial no. BL33 Skeleton L434	Young adult female Supine, extended Osteomyelitis/fracture of left ulna; degeneration of vertebrae	Late 3^{rd}-4^{th} c. AD Wooden coffin, layer of plaster Two greyware flasks; ceramic dish and bowl; coin (AD 323-324)	LTU434 material compacted within cranium NB: post-cranial cleaned

7.3 3 Results

7.3.3.1 Contaminants and ubiquitous lipid species

Many of the samples were only minimally soluble in the organic solvent indicating low levels of extractable lipids. All were contaminated by phthalate plasticisers, probably derived from the plastic bags in which they had been stored. Most contained little or no organic matter (#23) while the lipid species identified in the remainder (#13) were, in the main, of low abundance and widespread occurrence (see **Appendix 6.2**). The exception, from Armagh Road, E3 is discussed below (**7.3.3.2**).

Thus, most of the compounds present in these samples comprised ubiquitous end products of the degradation pathways of plant and animal tissues with some microbial input and so cannot be associated with any specific source. Indeed, their range and patterning suggests a variety of contributors. For example, LMM *n*-alkanes may represent a microbial or algal contribution while HMM compounds (>C_{23}) are generally considered to derive from the leaf waxes of higher plants (Kögel-Knabner 2002; Otto and Simpson 2006). Vegetative matter is also the most likely source of the commonly-occurring *n*-alkanols observed in many of the samples that provided positive results while the carboxylic acids (SFAs and MUFAs) could relate to microbial, animal and/or plant sources (Evershed 2008a; Jambu *et al.* 1993).

Sterols and stanols, where present, have greater diagnostic capability. Those identified confirmed inputs from both microbially-degraded plant (*β*-sitosterol; stigmastanol) and mammalian (cholesterol; 5α-cholestanol) tissues with the potential addition of faecal material (Bull *et al.* 2002; Evershed *et al.* 1997b). This combination of components is typical of the diverse lipids found in soil organic matter (Jandl *et al.* 2005; Kögel-Knabner 2002; Otto *et al.* 2005) and so cannot be considered of archaeological relevance. Nonetheless, some contribution from the decomposition of intrinsic deposits (e.g. floral tributes; food offerings) and/or the human remains (particularly in the residues associated with Burial 25, Great Dover Street and Skeleton 130, St Clare Street due to the broad range of cholesterol derivatives identified) could be present.

7.3.3.2 Site details and results: Armagh Road, Bow (eastern burial ground)

During 1972, development work on Armagh Road, Bow (AR 72), E3 (**Figure 7.8**) revealed four Roman period burials. Part of the eastern burial ground (**Appendix 6.2.3**), these consisted of a supine, extended adult female in a limestone sarcophagus accompanied by an iron key, and two pins, one of bone and the other of jet (Context 1). A second limestone sarcophagus with a fragment of lead attached to the lid was also discovered. This contained two individuals, an adult female whose remains were partially disarticulated (Context 4) and an adult male who was found supine, extended (Context 3). This relationship suggested that the sarcophagus had been re-opened in order to include the male who had a well-healed spiral fracture of the left tibia and fibula. The heavily truncated skeleton of a fourth individual, an adult female who had been interred in a wooden coffin, were found nearby (Context 2; Owen *et al.* 1973). Both of the sarcophagi contained a white substance "in a semi-liquid state" (Owen *et al.* 1973: 156-157). Analysis demonstrated that this material was fine-grained calcium carbonate, derived from a very pure marine limestone, possibly chalk (MacKenna 1972).

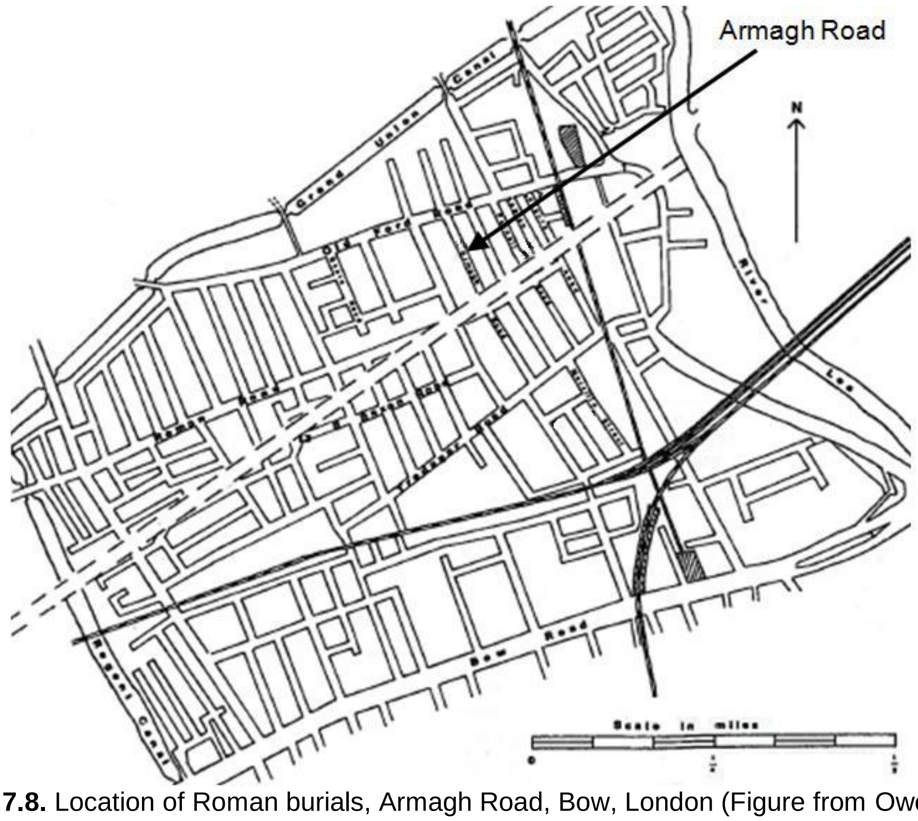


Figure 7.8. Location of Roman burials, Armagh Road, Bow, London (Figure from Owen *et al.* 1973: Figure 1, 135).

Adhering residues and materials associated with the near-complete individuals (AR72/1; AR72/3) interred within the limestone sarcophagi were analysed. These samples contained traces of *n*-alkanols and carboxylic acids in conjunction with a limited range of mammalian-derived steroidal compounds (cholesterol; 5α-cholestanol). In addition, an extensive homologous series of *n*-alkanes (C_{17-33}; maxima at C_{22}) and the isoprenoids pristane (Pr) and phytane (Ph) (**Figure 7.9a**) alongside regular steranes ($C_{27-29,}$ C_{28} dominant, ααβ more abundant), tricyclic polyprenanes (C_{23-25}) and a limited range of hopanes ($C_{27-30,}$ predominantly αβ) (**Figure 7.9b**) were identified from Context 1. Bacterial marker compounds, diploptene (hop-22(29)-ene, **Figure 7.10a**; as confirmed by comparison with the literature, **Figure 7.10b**) and diplopterol (hopan-22-ol) were also present in significant abundance. These were accompanied by traces of the triterpenoids, moronic acid (**Figure 7.11a**) and oleanonic acid (**Figure 7.11b**) and a derivative of the latter, 28-nor-olean-17-en-3-one (formed by decarboxylation followed by an hydrogen shift, Pastorova *et al.* 1998).

This combination of compounds is characteristic of bitumen/asphalt, the term used to describe "naturally occurring solid or liquid hydrocarbon deposits that are soluble in an organic solvent" (Killops and Killops 2005: 128). Petroleum products are of considerable commercial and archaeological significance. Extensive research has, therefore, been carried out with the aim of identifying and characterising natural ‘tar’ seeps and bituminous sediments (Connan 2012: passim). The main diagnostic components present in bitumen have been shown to be saturated hydrocarbons (*n*-alkanes), polyaromatic hydrocarbons (PAHs), tetracyclic steroid derivatives (e.g. steranes, diasteranes) and pentacyclic terpenoids (e.g. hopanes, oleananes, lupanes) (Hunt 1996: 64-104). The presence/absence and comparative abundance of many of these marker compounds and/or compound classes has also been shown to offer insights into the maturation stage of the bitumen and the source of contributions during its formation (marine, lacustrine or terrestrial organic matter) as well as providing indications as to the nature of the source rock (Connan *et al.* 2004; Hunt 1996: 380-393; Maurer *et al.* 2002).

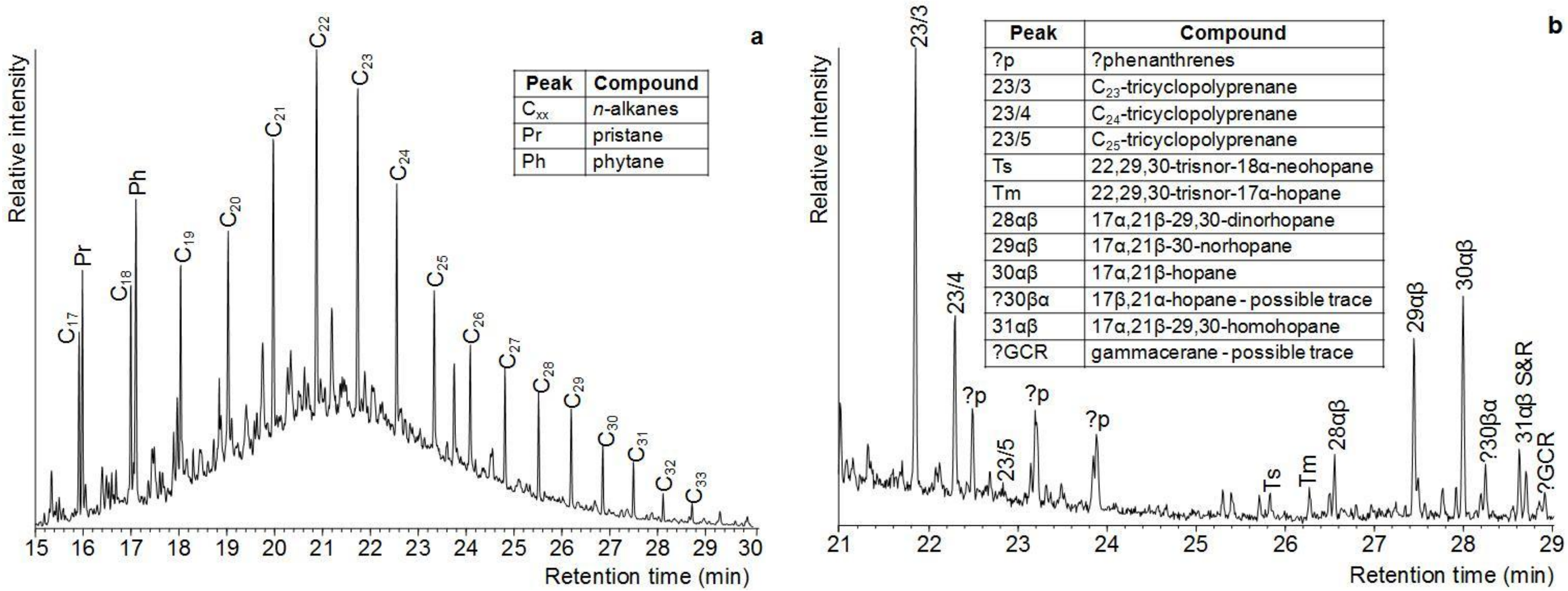


Figure 7.9. XICs, Context 1, Armagh Road, London, displaying diagnostic markers for the presence of bitumen: a. *m/z* 71, AR72-1a, homologous series of *n*-alkanes; b. *m/z* 191, AR72-1b, polyprenanes and hopanes.

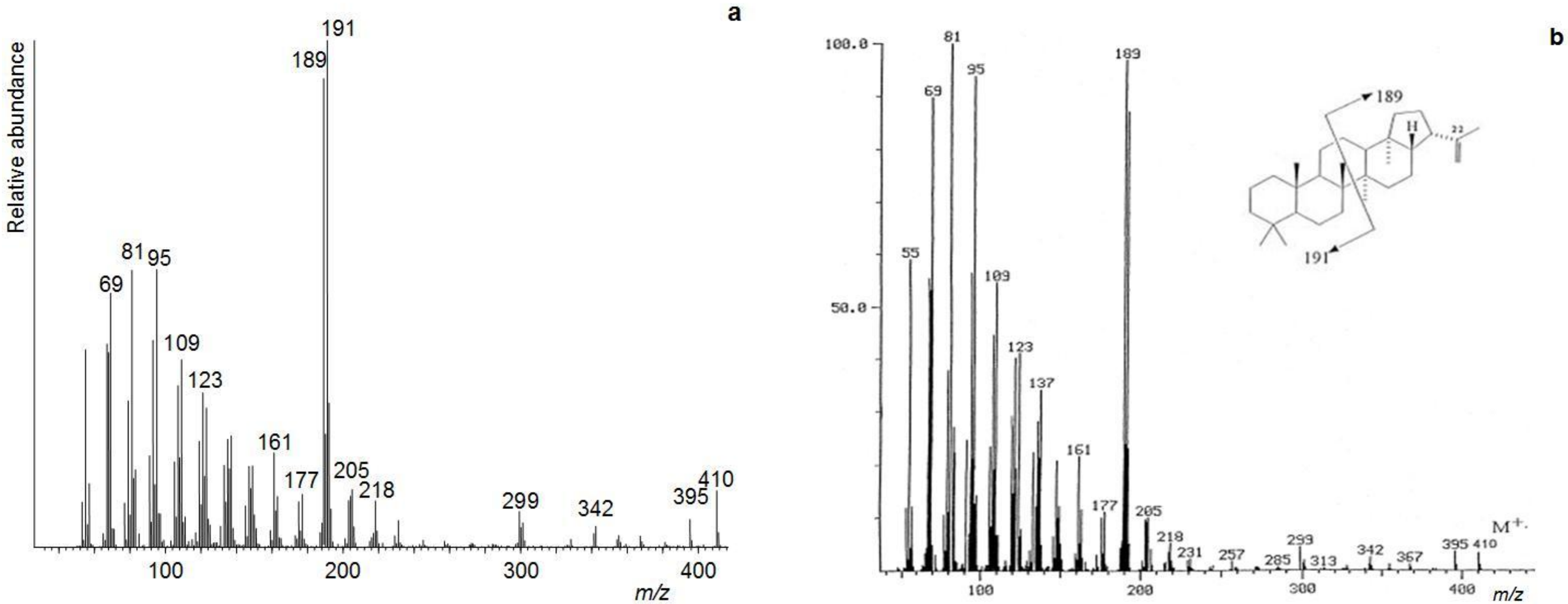


Figure 7.10. Mass spectrum assigned as hopan-22-ene (diploptene), a marker denoting bacterial degradation: a. 19.1 min, AR72-1a, Context 1, Armagh Road; b. extract from archaeological bone, femur, 9th-10th century AD (Figure from Evershed *et al.* 1995: Figure 7, 287).

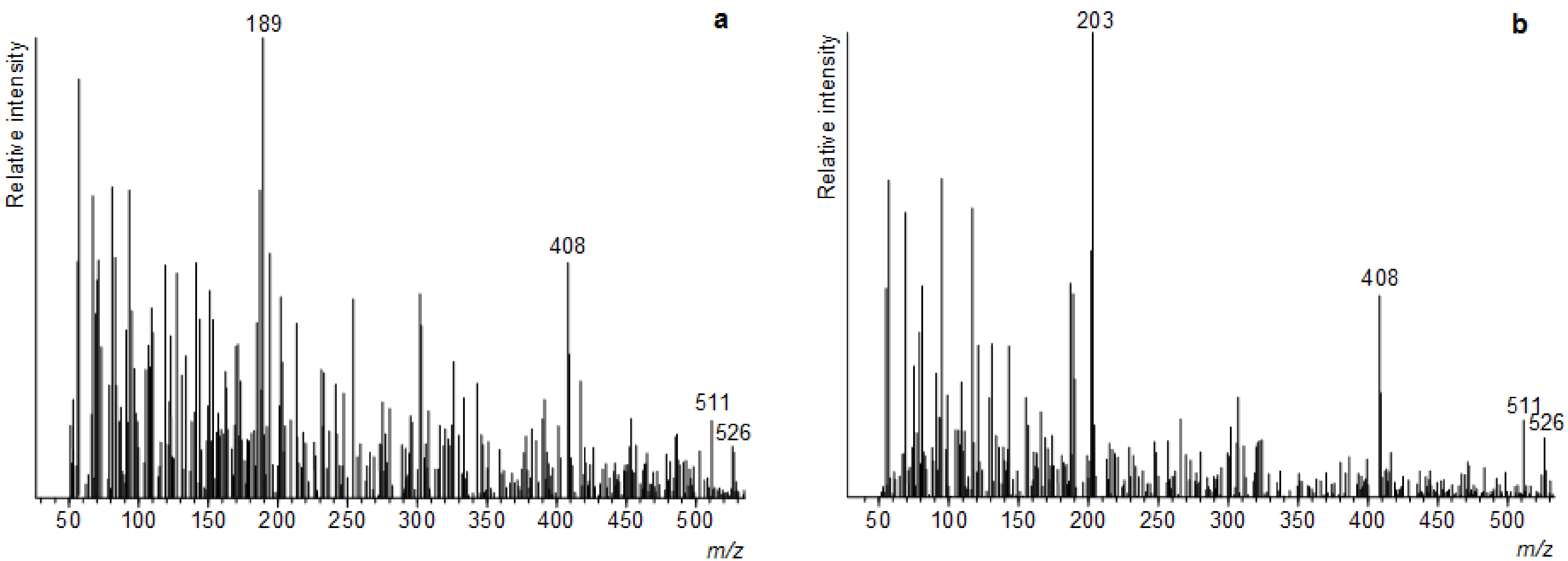


Figure 7.11. Mass spectra of triterpenoids, AR72-1b, Context 1, Armagh Road, assigned as: a. moronic acid; b. oleanonic acid.

In the samples from Context 1, Armagh Road many key marker compounds were not detected or of low abundance limiting comparison with published data. Nonetheless, the range (C_{17}-C_{33}) and relative abundances of the *n*-alkanes and isoprenoids (pristane and phytane) could be evaluated and was found to most closely resemble the patterning observed in lacustrine oils due to the smooth 'bell-curve' effect, even-over-odd preference and a pristine/phytane (Pr/Ph) ratio less than unity (**Table 7.4**). These factors suggest a mature extract with origins in an anoxic, reducing environment (Hunt 1996: 380; ten Haven *et al.* 1987) and are supported by the relationship between the C_{27-29} steranes with the dominance of the C_{28} isomers indicative of an algal input (Hunt 1996: 407, 555). Relatively high Pr/*n*-C_{17} and Ph/*n*-C_{18} ratios, the preferential loss of the C_{27} steranes and survival of only the more resistant phenanthrenes among the PAH components also seems to denote considerable biodegradation (Connan *et al.* 1992). Indeed, comparison of the Pr/*n*-C_{17} and Ph/*n*-C_{18} ratios with known source materials suggests a Type II kerogen seep (Hunt 1996: 542).

Table 7.4. Ratios (isoprenoids/*n*-alkanes) indicative of biodegeneration, Context 1, Armagh Road, E3.

Sample	Pristane/*n*-C_{17}	Phytane/*n*-C_{18}	Pristane/Phytane
AR72/1a	1.44	1.46	0.67
AR72/1b	1.19	0.70	0.77

The undisturbed nature of the deposits (despite damage to the lids of the sarcophagi during discovery) implies that this material could be of archaeological relevance. It is certainly an anthropogenic introduction as no natural deposits are present around London, although seeps have been recorded along the south coast (e.g. Sussex) (BGS 2011). Moreover, in antiquity, bituminous materials from the Dead Sea and surrounding regions of the Levant have been shown to have been widely utilised and traded (Connan 2012: 148-154; Connan *et al.* 1992). These substances were employed as caulking materials, adhesives, sealants, in medicines and Egyptian embalming processes, although generally as a minor component in the latter instance (e.g. Colombini *et al.* 2000; Nissenbaum and Buckley 2013; Harrell and Lewan 2002; Maurer *et al.* 2002). Unfortunately, in relation to these samples from Armagh Road, contamination as a result of the work carried out by the Water Board (which revealed these burials) or ingress/seepage from activities such as road surfacing is a strong possibility (although a sample of modern bitumen employed in this manner was found to contain very few solvent-extractable compounds, presumably as a result of efficient processing methods). Thus, although further analysis (e.g. using geochemical techniques to isolate the bound, asphaltene, fractions and/or isotopic characterisation) might be able to help identify the source region (c.f. Connan 2012: 90-134), the key question of a Roman or modern date for its introduction to Britain may remain elusive.

The other hopane-based compounds identified, diploptene and diplopterol, are also widely found in oil-containing sediments where they act as indicators of inputs from terrestrial sources (e.g. Brault and Simoneit 1988; Prahl *et al.* 1992). Excreted by hopane-producing bacteria, they could, however, equally derive from the bacterial degradation of bone and, as such, have been identified in archaeological burial contexts (e.g. Evershed *et al.* 1995). In this instance, since these moieties were not found in any of the other samples from Roman London, a relationship with the bitumen seems the simplest explanation.

The source of the triterpenoids, moronic acid, oleanonic acid and the degradation product 28-norolean-17-en-3-one is, likewise, difficult to ascertain. Background levels of triterpenic compounds including oleananes are often reported in sediments as the result of inputs from higher plant matter and so are often present in terrestrially-derived petroleum products (Hunt 1996: 95-97). A flowering angiosperm contribution to the organic matter within the sarcophagus as part of the bitumen, due to the deposition of floral tributes or soil ingress is perfectly possible. Nonetheless, this combination of resin acids does not occur in native British species and has not, to my knowledge, been reported in petroleum products (although it should be noted that the literature is vast). These compounds are, however, considered biomarkers for *Pistacia* spp. resins with 28-norolean-17-en-3-one a significant degradation product denoting natural aging and/or thermal-alteration (Colombini *et al.* 2000; Stern *et al.* 2003). Thus, the body of this individual may have been treated with a combination of plaster and bitumen with the possible addition of mastic/terebinth resin.

7.3.4 Summary and interpretation of findings

Analysis of samples recovered from numerous late Roman period inhumations from the burial grounds of *Londinium* (with the exception of the 'Spitalfields Lady', see **7.4**) provided no clear evidence for the application of plant exudates, even when plaster was present. Evaluation of the presence/absence of resinous substances was, however, severely limited by the nature of the available samples and attendant taphonomic considerations. Organic materials, even those containing water-insoluble, degradation resistant compounds such as HMM terpenoids, may simply not be recoverable particularly if they were deposited in low abundance and/or with individuals interred in wooden coffins (discussed in **8.5**).

The data obtained from the grave deposits associated with the adult female interred in a sarcophagus in the eastern burial ground (Context 1, Armagh Road, E3) is, therefore, tantalising. Both *Pistacia* spp. resins and bitumen have been attested as part of Egyptian embalming materials (e.g. Buckley *et al.* 2004; Connan 2012: 177-182; Maurer *et al.* 2002; Serpico 2000) although this combination has not, previously, been identified in Roman period burials elsewhere in the Empire. Nonetheless, as contamination cannot be ruled out, the archaeological relevance of this find cannot be confirmed.

7.4 Case Study 3: 'Spitalfields Lady', London, E1 (Brettell *et al.* 2015a)

7.4.1 Background

The northern cemetery of Roman London was located outside the city walls along the line of Ermine Street (Bishopsgate) in the area that now comprises Finsbury Circus, Liverpool Street Station and Spitalfields Market, London, E1 (Evans and Pierpoint 1986; **Figure 7.12**). During re-development in the late 1990s-early 2000s, excavations carried out around Bishopsgate and Spitalfields (SRP 98) revealed over 100 Roman period burials (Swift 2000; Thomas *et al.* 2003). Five of these, aligned at 90° to the road on a slight rise, had been interred in more substantial containers. Three of these consisted of sarcophagi which had been robbed in antiquity, another contained the remains of a child encased in plaster (not available for sampling) within a timber-lined mausoleum and accompanied by eight glass vessels. The fifth was complete and undamaged (Thomas 1999; Thomas *et al.* 2003).

Figure 7.12. Roman *Londinium* superimposed on modern London showing the northern burial area along Ermine Street and the location of find (SK15903) known as the 'Spitalfields Lady' (MoL 2014).

Dated to the mid-4th century AD, this substantial sarcophagus was made of Barnack stone (limestone) from Cambridgeshire and enclosed a still-sealed elaborately decorated lead coffin. Meticulous laboratory excavation showed this to contain the well-preserved skeleton of a female in her early twenties (**Figure 7.13a**). No signs of trauma or disease were observed on her bones and her dentition was in good condition. Dubbed the 'Spitalfields Lady', she had been laid supine, extended with head to the west. Textile fragments and bay leaves, possibly representative of a pillow, were found below her head while further textiles, identified as silk damask (obtained from China and probably woven in Syria) and wool, together with spirally spun gold thread were found within the silt beneath the skeleton. This suggests that the young woman had been clothed in a gold-embroidered silk damask garment and wrapped in woollen cloth (Museum of London 1999; Thomas 1999).

Grave goods were also present at the foot of the coffin. These consisted of two high quality imported glass phials (one between the lead and stone containers), a jet rod, other jet artefacts and a small cylindrical box. It has been suggested that these items could represent cosmetic equipment including unguent or perfume containers (Museum of London 1999; Thomas 1999). Isotope analysis indicated that the lead of the coffin derived from UK sources but the pathologically high lead isotope values in the dentition of the 'Spitalfields Lady' came from a non-English source. Strontium ratios indicated a childhood spent on Mesozoic sediments (widespread in south-east England and across continental Europe) with the combination of lead and strontium values very similar to those of three individuals from Rome. Thus, it seems that she may have been born abroad, perhaps in Italy, and have migrated to London shortly before her death (Montgomery *et al.* 2010).

Figure 7.13. The 'Spitalfields Lady', SK15903, Bishopsgate, London: a. newly opened lead coffin and sarcophagus showing grave deposits surrounding skeletal remains (provided by MoL for author to use in publications, ©MoL); b. F290, flot sample; c. coloured solvent extract obtained from F290; d. PSA292, material from between the lead coffin and the sarcophagus; e. residue adhering to the hyoid bone; f. P26, sample from within the lead coffin, cranial region, showing orange flecks; g. P12, sample from 'gut' region (Author).

7.4.2 Sample selection

Three samples were initially collected with the kind permission of the MoL, facilitated by Dr Rebecca Redfern. These consisted of a dark residue adhering to the inner, concave surface of the hyoid bone (**Figure 7.13e**) and yellow-orange residues from areas of the skeletal remains (e.g. hand and foot bones) that had not been scrubbed clean for display alongside small amounts of associated debris from within the plastic bags in which they had been stored. Grave deposits were also requested as ‘environmental’ samples were recorded as having been collected during excavation. Earmarked for disposal, these were subsequently located and found to comprise sediments from within the lead coffin (Context 15904), selected for pollen analysis, and from between the coffin and the stone sarcophagus (Context 15900), collected for particle size analysis. Although this work had not been carried out some portion of the latter had been flotated, possibly in industrial methylated spirits (IMS), which may have removed some, if not all, organic solvent-soluble lipid components and introduced contaminants.

The remainder, however, were untreated and provided a series of spatially distributed samples (#9) from around the skeletal remains (denoted as P) and five control samples (PSA) from between the lead coffin and stone sarcophagus as some soil ingress was posited by the excavators (Thomas 1999). In addition, a decision was made to process the flot (F) samples in order to compare results with those from their corresponding PSA samples and to ascertain the value of materials treated with IMS in respect of pursuing analysis of those from St Martin-in-the-Fields (**7.3.3.1**). Sample images, TICs and additional data are provided in **Appendix 6.3; Disc 1, File 6.3.**

7.4.3 Results

7.4.3.1 Initial findings (samples associated with the skeletal remains)

The yellow-orange residues on limited areas of the skeleton (e.g. foot and hand bones) had the appearance of soil marks while the associated debris had a sandy, gravel-like texture and seemed to have been rinsed in water. All of the samples were minimally soluble in the organic solvent and contained low levels of lipids. The yellow-orange residue (SPF OR) was found to contain traces of *n*-alkanes (C_{18}-C_{22}) and *n*-alkanols (C_{14}-C_{18}). Likewise, the compounds present in the coffin debris (SPF BD1) consisted of a range of *n*-alkanes (C_{17}-C_{26}), *n*-alkanols (C_{10}-C_{18}) and traces of SFAs ($C_{12:0}$-$C_{16:0}$). The most prominent peaks were found to be phthalate plasticisers and other contaminants, which probably derived from the plastic storage bags.

In contrast, the dark residue from the inner surface of the hyoid contained low levels of plasticisers with no *n*-alkanes or *n*-alkanols observed. The main compounds were SFAs ($C_{8:0}$-$C_{16:0}$, $C_{18:0}$) and triterpenic moieties (**Figure 7.14; Table 7.5**). The latter were present in low abundance and so definitive identification proved problematic, although the compounds appeared to have pentacyclic oleanane skeletons based on their characteristic fragmentation patterns (Assimopoulou and Papageorgiou 2005a).

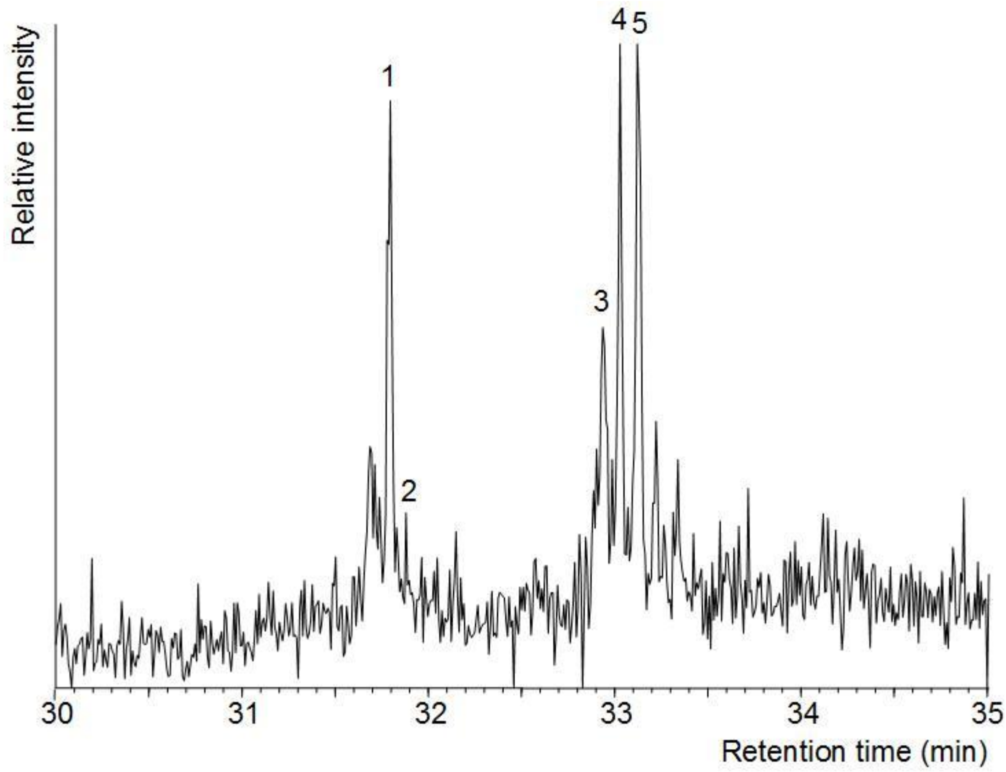


Figure 7.14. Partial XIC (*m/z* 203), residue from hyoid bone, SK15903, SRP 98, London. Peak identifiers relate to **Table 7.5**.

Table 7.5. Provisional identification of triterpenic compounds (TMS derivatives), residue, hyoid bone. SK15903 based on molecular ion (M^{+}.), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
1	498	218	483, 279, 203>189, 73	β-amyrin
2	410	163	281, 253, 218, 207, 191, 175, 133	28-norolean-17-en-3-one
3	526	189	511, 409, 320, 203, 133, 119	?moronic acid
4	526	203	511, 408, 320, 307, 219, 189, 133	oleanonic acid
5	438	203	409, 232, 189, 175, 133	oleanonic aldehyde

The mass spectra obtained were tentatively identified as denoting the presence of β-amyrin, 28-norolean-17-en-3-one, oleanonic acid and oleanonic aldehyde. These compounds are found in exudates from genera within the Burseraceae, Anacardiaceae and Hammamelidaceae families (Assimopoulou and Papageorgiou 2005a; Modugno *et al.* 2006b; Stacey *et al.* 2006). In addition, the minor peak eluting prior to oleanonic acid appeared to be moronic acid, characteristic of resins from the genus *Pistacia*, although other biomarkers such as masticadienonic acid and *iso*masticadienonic acid were not observed. Locating the 'environmental samples' reportedly collected during excavation was, therefore, deemed important in order to provide confirmation of these findings.

7.4.3.2 Samples between the sarcophagus and coffin (Context 15900)

The flot samples appeared to consist of gravel, sand and fragments of vegetation devoid of any soil organic matter (**Figure 7.13b**). During solvent extraction a violet colour was immediately obvious in sample F290 (**Figure 7.13c**) and a blue-black colour became apparent in F292b, F293 and F294 during extract concentration. There are a number of possible sources for this colouration including:

- the synthetic aniline dye added to methylated spirits that are available to the general public and which could have been used, during processing, in the flotation of these samples in the absence of industrial methylated spirits (IMS);
- the synthetic dyes present in the inks of marker pens as labels, with sample details inscribed on them, may have been placed within the sample bags although there is no current evidence of this;
- the presence of a natural dye (e.g. Tyrian purple), or dye mixture (e.g. madder and indigo) of archaeological origin which could have been scattered over the outside of the lead coffin or used to colour a cloth that had been draped over the coffin which had subsequently decayed. Such evidence has been recovered from a number of Roman period burials (Wild 2013).

A portion of each of the flot samples was, therefore, retained and stored in the absence of light for further analysis. It should be noted that the reportedly untreated particle size analysis (PSA) samples obtained from the same contexts did not produce any visible coloration during solvent extraction. These comprised clods of dark brown soil with inorganic inclusions (e.g. stones, mollusc shell fragments).

The results of the GC-MS analysis also provided unexpected results as, despite their smaller mass and treatment with IMS, the flot samples incorporated more lipid moieties, although in very low abundance, than their corresponding PSA samples (**Figure 7.13d**). Indeed, the latter contained little except phthalate plasticisers. These modern contaminants were also observed in the flot samples. Where other lipids were present, in both sets of samples, these consisted of low levels of ubiquitous *n*-alkanes ($C_{15\text{-}33}$), *n*-alkanols ($C_{12\text{-}30}$) with a bimodal distribution, SFAs ($C_{12\text{-}18}$), sterols (cholesterol and β-sitosterol) and stanols (5α-cholestanol and stigmastanol). These compounds are characteristic of degraded plant and animal matter and could derive from extrinsic (soil ingress) or intrinsic (funerary deposits) sources. Traces of triterpenoids denoted by fragment ions at *m/z* 189 and 203, one peak with a molecular ion at 526, were also observed in F292a and suggest the presence of a plant resin similar in nature to that indicated during analysis of the residue from the hyoid bone (**7.4.3.1**).

It is interesting to note, therefore, that one of the glass phials was found between the lead coffin and the sarcophagus (Thomas 1999) as this raises questions about its relationship with the possible dye and resin traces. Sampling the inner surface of the vessel for such evidence was not possible, however, as the glassware had been cleaned and placed on display soon after discovery.

7.4.3.3 Samples from within the lead coffin (Context 15904)

No such ambiguity pertained to the silt samples obtained from within the lead coffin (**Figure 7.13f-g**). These fully contextualised, spatially distributed samples contained phthalate plasticisers and a range of common lipid species indicative of an input from degraded plant and animal matter. In addition, suites of both diterpenic and triterpenic compounds were identified. The diterpenoids were observed in all of the samples with the exception of those from between the arm bones and sides of the lead coffin (P22 <335>; P24 <337>) and were found to have abietane and pimarane skeletons. Those present comprised the resin acids, pimaric (PM), sandaracopimaric (SDPM), *iso*pimaric (IPM) and abietic (AB) acid, and early stage degradation products of the latter, dehydroabietic (DHA) and didehydroabietic (DDHA) acid (**Figure 7.15; Table 7.6**).

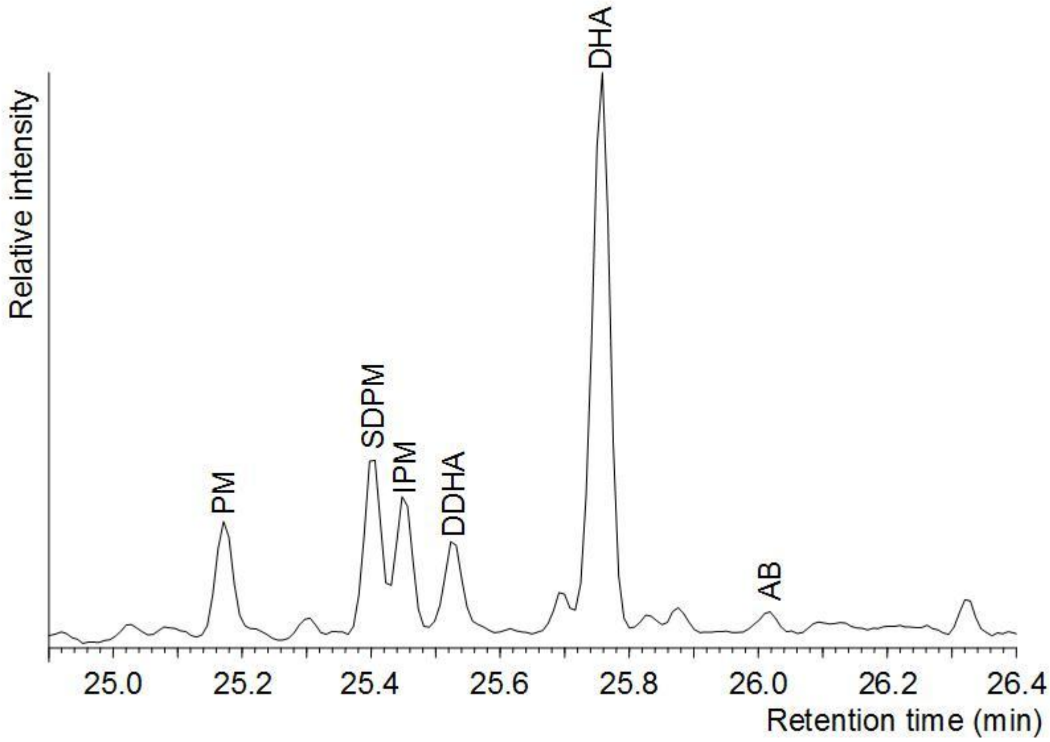


Figure 7.15. Partial TIC (24.8-26.4 min) P12, grave deposit, near the feet, SK15903, SRP 98, London. Peak identifiers relate to **Table 7.6**.

Table 7.6. Identification of diterpenoids (TMS derivatives) in the grave deposits, lead-lined coffin. SK15903, SRP 98, London based on molecular ion ($M^{+\bullet}$), base peak (BP) and key fragment ions.

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
PM	374	73	121, 133, 191, 207, 257, 299, 359	Pimaric acid
SDPM	374	121	73, 91, 143, 241, 257, 359	Sandaracopimaric acid
IPM	374	241	73, 105, 143, 256, 257, 359	Isopimaric acid
DDHA	370	237	73, 103, 143, 195, 209, 252, 355	Didehydroabietic acid
DHA	372	239	73, 129, 143, 171/3, 185, 240, 255	Dehydroabietic acid
AB	374	256	73, 105, 185, 213, 241, 257	Abietic acid

These compounds are biomarkers for conifer resins from the Pinaceae family with the relative abundance of pimaric acid and derivatives of abietic acid similar to those observed in aged *Pinus* spp. resins (c.f. Colombini and Modugno 2009; **5.3.2**). The survival of the precursor acids and absence of defunctionalised compounds indicative of more extensive degradation processes (e.g. norabietadienes, norabietatetraenes) demonstrates that the resin was relatively well preserved. Likewise, highly resistant end products indicative of the thermal processing of Pinaceae resins or resinous woods were not present (e.g. methyl dehydroabietate (MDHA) and retene (RET)).

Confirmation of the botanical nature of this resin was made through comparison with modern Pinaceae products, including *Pinus pinaster* and *Pinus sylvestris* resins and a *Pinus sylvestris* tar (**Appendix 3.1**). The compounds present in the archaeological samples displayed identical mass spectra to key biomarkers in the reference materials. Moreover, the absence of indicators of extensive thermal alteration (i.e. MDHA, RET and neutral intermediates) in the 'Spitalfields Lady' samples is in clear contrast to the range of moieties in the *P. sylvestris* tar.

In addition, triterpenic compounds were present in all of the grave deposit samples from within the lead coffin, although those on the outer side of the arms (P22<335>; P24<337>) were again differentiated as they contained only trace levels of degradation resistant neutral compounds. These terpenic moieties were found to have oleanane and tirucallane skeletons based on their characteristic fragmentation patterns with key components identified as moronic (MA), oleanonic (ONA), *iso*masticadienonic (IMDA) and masticadienonic (MDA) acid, respectively (**Figure 7.16; Table 7.7**). These precursor acids are biomarkers for resins from the genus *Pistacia* (Barton and Seoane 1956; Caputo *et al.* 1978; Papageorgiou *et al.* 1997; **5.3.3**) whose survival demonstrates that the resin is relatively well preserved. This is confirmed by the absence of ocotillone-type molecules (oxidised dammaranes) which are often prevalent in aged *Pistacia* spp. resins. Moreover, the low abundance of 28-norolean-17-en-3-one (28NO17) and related neutral derivatives suggests that the resin is unlikely to have been heated (at least, not to any significant extent) prior to deposition.

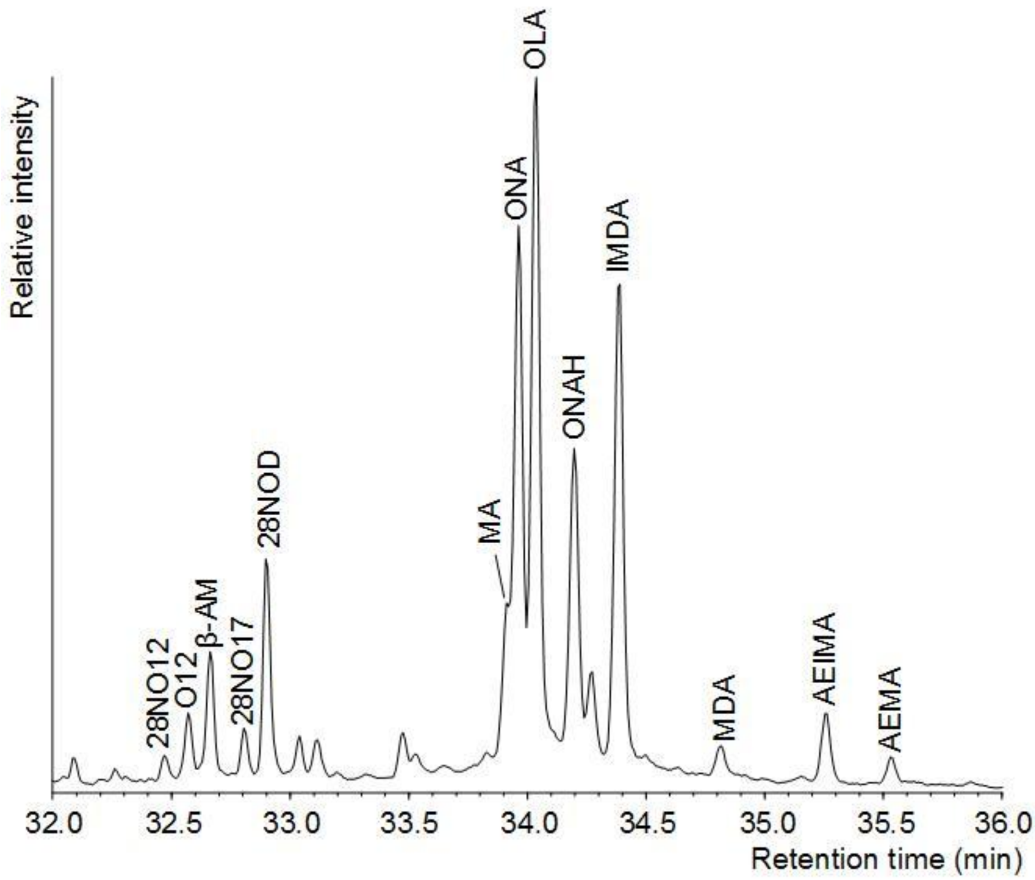


Figure 7.16. Partial XIC (*m/z* 203) P25, grave deposit, left of cranium, SK15903, SRP 98, London. Peak identifiers relate to **Table 7.7**.

Table 7.7. Identification of triterpenic compounds (TMS derivatives), grave deposits, lead-lined coffin, SK15903, SRP 98, London based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
28NO12	410	204	395, 381, 313, 245, 215, 189, 175, 133	28-norolean-12-en-3-one
O12	424	218	409, 218, *203 much>189,* 122, 95, 55	olean-12-en-3-one (β-amyrenone)
β-AM	498	218	483, 409, 393, 279, *203 much>189*, 190	β-amyrin
28NO17	410	163	393, 279, 257, 218, 203, 133, 119	28-norolean-17-en-3-one
28NOD	408	408	379, 339, 229, 216, 203, 189, 173, 129	28-norolean-12,17-dien-3-one
MA	526	189	511, 409, 391, 320, 307, 219, 203, 133	moronic acid
ONA	526	203	511, 408, 393, 320, 307, 219, 189, 133	oleanonic acid
OLA	600	203	585, 510, 482, 392/3, 320, 279, 189, 133	oleanolic acid
ONAH	438	203	409, 320, 232, 189, 175, 133, 119, 105	oleanonic aldehyde
IMDA	526	511	421, 393, 307, 257, 243, 213, 185, 169	*iso*masticadienonic acid
MDA	526	511	421, 393, 311, 257, 213, 185, 169, 143	masticadienonic acid
AEIMA	570	495	555, 511, 423, 393, 241, 213, 189, 169	3α-acetoxy-3-epi*iso*masticadienolic acid
AEMA	570	495	555, 511, 423, 405, 241, 213, 185, 169	3α-acetoxy-3-epimasticadienolic acid

Further confirmation was made through comparison with a range of modern *Pistacia* spp. exudates (**Appendix 3.2**). A clear correspondence can be demonstrated between the mass spectra of the biomarkers in these modern resins and those in the archaeological samples.

7.4.4 Summary and interpretation of findings

In the case of the ‘Spitalfields Lady’ (SRP 98, SK15903) chemical evidence for two different resins was obtained through analysis of silt-like grave deposits from the base of the inner lead coffin. The presence of a relatively well preserved conifer resin derived from a member the Pinaceae family, possibly *Pinus* spp., was denoted by pimaric acid and its isomers together with abietic acid and its early stage derivative, dehydroabietic acid. The highly persistent end stage products of the degradation pathways of these diterpenoid resin acids were not observed which rules out extensive heating either of a Pinaceae resin or resinous wood to produce a tar or pitch although gentle warming remains a possibility. In addition, these samples contained moronic, oleanonic, masticadienonic, *iso*masticadienonic acid and their derivatives, biomarkers for resins from the genus *Pistacia*. The range and relative abundances of these triterpenic compounds again indicates good preservation while the low abundance of proposed markers of heating (e.g. 28-norolean-17-en-3-one) suggests that the resin had been deposited in its natural state.

The most parsimonious explanation is, therefore, that a mixture of unheated *Pistacia* spp. and Pinaceae resins were used as part of the mortuary rites afforded the ‘Spitalfields Lady’. Moreover, the multiple samples obtained from different areas of the inner coffin allowed the patterning of these exudates in relation to the human remains to be considered (**Table 7.8**). Biomarker compounds for both resins were found to be present along the entire length of the coffin (head to feet) with a greater abundance in the midline and only minimal traces in the samples between the arms and sides of the coffin (P22<335>; P24<337>). This suggests an intimate association with the body and/or textile wrappings. In addition, the triterpenoids denoting *Pistacia* spp. resins appeared more prevalent than the Pinaceae resin diterpenoids in the upper torso (P23<336>) and cranial (P25<338>; P26<339>) area while the latter were more plentiful in the pelvic region (P12<295>, pelvis/‘gut’; P21<334>, right femur). Further evaluation using an internal standard is, however, required to confirm any such indications.

Table 7.8. Distribution of the two resins identified based on a rough comparison (using manual integration) of the relative abundance of diagnostic compounds in each chromatogram: *very low <50000; **low 50001-20000; ***moderate 20001-40000; ****high 40001-60000; *****very abundant 60001^+.

Sample/context	Area of body	PM	DHA	MA	ONA
P26<339>	Right of cranium	*	**	****	*****
P25<338>	Cranial area (left)	**	**	*****	*****
P23<336>	Upper body	*	**	***	***
P24<337>	Right arm	*	*	*	*
P22<335>	Left arm	*	*	*	*
P12<295>	Pelvis 'gut'	****	*****	**	***
P21<334>	Right femur	***	****	**	***
P16<329>	Left lower leg	**	*	*	*
P15<298>	Foot of coffin	**	*	***	***

It should also be noted, that the results obtained from the grave deposits provide credence to the vestiges of a *Pistacia* spp. resin (although not Pinaceae, which may support the posited higher abundance of *Pistacia* spp. in this area of the body) initially recovered from the residue adhering to the hyoid bone. This is of some significance in terms of the confidence that can be placed in trace findings when grave deposits are no longer extant for analysis. Likewise, the triterpenic compounds in the flot sample from between the lead coffin and stone sarcophagus, in the vicinity of the glass vessel, may indicate that this was the container in which the *Pistacia* spp. resin had been transported although definitive evidence is lacking.

7.5 Case Study 4: Lead-lined burials, Winchester, Hampshire

7.5.1 Background

Winchester (*Venta Belgarum*), Hampshire, UK (SU 485 295) shows evidence of Roman occupation from c. AD 50-70 with drainage works and the construction of a defensive circuit followed by the laying out of a formal grid-pattern of streets and public buildings in the late 1st century AD (Qualmann *et al.* 2012). Situated at an important road junction and river crossing, the Roman town became the *civitas* capital for the region. Its increased prosperity in the 2nd-early 4th centuries AD was marked by further groundworks, the addition of a masonry wall to strengthen the defences and a number of substantial town houses containing mosaic floors and hypocausts. As elsewhere, after c. AD 350, this prosperous settlement underwent a significant period of change and effectively ceased to function by the early 5^{th} century AD (Booth *et al.* 2010: 523-526; Cunliffe 1964; Qualmann *et al.* 2012; Scobie *et al.* 2008).

Following Roman law, a number of burial grounds existed outside the town walls to serve its inhabitants and, perhaps, those dwelling in its immediate hinterland (Qualmann *et al.* 2012). The best known is the extensive burial area that developed outside the North Gate of the town along the eastern side of the road to Cirencester. In use from the late 1^{st}-4^{th} centuries AD, it extended from Victoria Road East as far as Lankhills (Booth *et al.* 2010; Clarke 1979; Ottaway and Rees 2012; Scobie *et al.* 2008). A similar substantial mixed cremation and inhumation burial ground also appears to have existed to the south of the town (Qualmann *et al.* 2012). In the late 3^{rd}-early 4^{th} century AD, new burial areas were established to the west of the Cirencester Road in the vicinity of Victoria Road West and then Andover Road (Ottaway and Rees 2012; Teague 1999) and in the suburbs beyond both the western and eastern gates of the town. These demonstrate considerable use into the late 4^{th} century AD and possibly beyond (Collis 1978; Morris 1986; Ottaway and Rees 2012; Scobie *et al.* 2008).

At Winchester, cremation formed the principal Roman period rite until the mid 3^{rd} century AD, with the exception of infants under c. eighteen months old who were often inhumed (Ottaway and Rees 2012). This pattern is most clearly demonstrated in the northern burial area where, in earlier phases of use, cremated remains were interred in simple urns or inhumation-sized pits. By the beginning of the 4^{th} century AD, however, the predominant mortuary rite had changed with the late Roman burial grounds established at Lankhills and to the west of the Cirencester Road found to house over 800 inhumations, the majority in coffins and accompanied by grave goods (Clarke 1979; Booth *et al.* 2010). Considerable variety within these parameters has been noted (Baldwin 1985; Ottaway and Rees 2012) and suggestions have been made for the presence of intrusive, foreign elements in the burial population at Lankhills (Clarke 1979: 364-367). This may be supported by the presence of 3^{rd}-4^{th} century Germanic-style military equipment, imported artefacts and dental enamel isotope ratios indicative of a diversity of origins (Cool 2010; Chenery *et al.* 2010; Eckardt *et al.* 2009; Evans *et al.* 2006).

Grave goods denoting age, gender and status distinctions, although not great wealth, were identified from both the northern and eastern cemeteries with some artefacts indicative of a mixture of classical and native religious beliefs, perhaps with an emphasis on the horse-goddess Epona (Booth *et al.* 2010: 516-519; Ottaway and Rees 2012; Rees *et al.* 2008: 389-390). It has also been proposed that St Martin's Close to the east of the town may fit the parameters for a late Roman Christian cemetery with organised, west-east aligned, generally unaccompanied burials many of whose body positions suggest that they were wrapped in shrouds (Woodward 1993: 236-237; Ottaway and Rees 2012; Sparey Green 2003). The site certainly displays a different character to the other eastern concentration of burials at Chester Road. This may, however, simply be a question of chronology as Chester Road appears earlier in date, although simultaneous use by population sub-groups is equally possible (Booth *et al.* 2010: 531; Rees *et al.* 2008: 389).

Despite this variety of rites, no stone sarcophagi have been found and only five individuals interred in lead-lined coffins identified, three from St John's Church (excavated in the 1800s), one from St Martin's Close (F57) and one from the site of the old Eagle Hotel, Andover Road (G336) (Ottaway and Rees 2012). Of these, only F57 and G336 were found to be extant.

The first, Burial F57, was recovered in 1985 during renovation works at St Martin's Close, Winnall to the east of Winchester. This badly damaged, lead-lined coffin contained the remains of a young adult female (c. 25-35 years old) placed supine with head to the west and was one of a pair (with F50, an elderly female) located below what appeared to be a revetted mound of redeposited chalk. Wood-staining and large iron nails with adhering wood fragments indicated the presence of an outer coffin made of ash. A small tile cist was noted at the foot of the grave with iron nails suggesting the presence of a box. Another cist, denoted by tiles and painted wall plaster, and associated with fragments of animal and bird bone was found near the head of the coffin (Morris 1986; Ottaway *et al.* 2012). Loose plaster packing was observed around the body. This, together with plaster from the smaller of the tile cists, was analysed using X-Ray Diffraction (XRD) and found to be gypsum (Ottaway *et al.* 2012). A composite, double-sided, antler comb dated to the late 4th century AD had been placed near the left shoulder of this individual (Cool 2010: 272-273; Rees *et al.* 2008: 64-66) while copper alloy sheeting and iron nails, possibly representative of a box, and some fine gold thread were also found within the coffin. The latter suggests that the body may have been richly dressed or wrapped in an embroidered shroud (Rees *et al.* 2008: 296). The condition of the skull, which was visibly more degraded than the remainder of the skeleton, led to a suggestion the head had received differential treatment (Morris 1986).

The second burial sampled (G336) was recovered from the old Eagle Hotel site, Andover Road, Winchester in 1998 (Teague 1999) and featured in the BBC programme 'Meet the Ancestors' (Richards 1999: 84-107). One of around forty-eight late Roman graves uncovered, G336 was orientated north-south, in contrast to the remainder of the burials, and appeared to have been the primary inhumation. This robust adult male had been interred in a lead-lined oak coffin within a particularly deep grave cut. Some soil ingress was noted but, otherwise, the burial appeared undisturbed. Many of the skeletal elements were fragile, contained extensive brushite crystal deposits within the trabecular bone and were marked by dark stains and residues although the cranium was well preserved. Osteological analysis indicated that this individual, around 35-45 years old at death, had been placed supine, in a fully extended position and may have been wrapped in a shroud as fragments of mineralised fabric (linen or wool) were found at the foot of the coffin (Teague 2012). A coin of the Emperor Constantine, dated AD 316-317 was also found adhering to the base of the coffin near the right hand (Richards 1999: 98-100). Isotope ratios present in the dentition were consistent with an origin in southern Britain (Montgomery *et al.* 2012: 127).

Table 7.9. Details of samples collected from the burial grounds around Winchester, UK (for images, TICs and tabulated results see **Appendix 6.4**).

Site location	Site information	Burial id.	Osteological details	Coffin, fill, grave goods	Sample information
SU 4882 2972; Morris 1986; Ottaway *et al.* 2012; Rees *et al.* 2008					
St Martins' Close Winnall Winchester, UK	*Located:* east of Winchester, on the western slope of St Giles' Hill *Recovered:* c. 50 inhumation burials; masonry structure, 2 burials, 1 in lead-lined coffin; 9/10 with packing, lining or covering of stone slabs/tiles. *Observations:* WE aligned; some ?enshrouded; minimal grave goods	F57	Adult female c. 25-35 years Supine WE aligned (head to west) One of a pair with F50, mature adult female, below a revetted mound of redeposited chalk.	Late 4^{th} c. AD Ash wood coffin Lead-lined, gypsum packing Within mausoleum/walled enclosure Two tile cists: boxes (nails); painted wall plaster; animal and bird bones. Grave goods: composite, double-sided antler comb; copper sheeting; nails; fine gold thread	Residues: • F57S, from scapula • F57M, from mandible Associated materials: • F57R, orange fragments (?inorganic) Found mixed with molluscan remains, burnt mammal bone and bird bones (?from tile cist)
SU 4791 2996; Richards 1999: 84-107; Teague 1999, 2012					
Eagle Hotel (closed) Andover Road Winchester	*Located:* outside the North gate; west of the road to Cirencester *Recovered*: evidence of c. 40 inhumation burials; some prone; 1 decapitation; 1 lead-lined; all ages; both sexes *Observations*: WE aligned; arranged in rows; few grave goods; 4^{th} century AD coins; some ?enshrouded; disturbance from Saxon pits	G336	Robust adult male c. 35-45 years Supine, extended NS aligned ?the primary inhumation Cranium well preserved Remainder of the skeletal elements in poor condition – extensive brushite formation	Early 4^{th} c. AD Oak wood coffin Lead-lined Textiles: mineralised linen or wool Grave goods: coin of Constantine (AD 316-317)	Residues: • G336F, from left femur Fill (control samples): • 7 samples of 'contaminated' fill from above lid • 1 sample of chalk rubble from grave cut Grave deposit samples: • fragmented bone and associated materials (x4) • light fraction of flot • 1mm fraction of sieved material • material with lead fragments from base of coffin • extract from mineralised textiles

7.5.2 Sample selection

In total, twenty-one samples from burial F57, St Martin's Close and G336, the focal burial at the Eagle Hotel site were analysed (**Table 7.9**). The skeletal remains of both individuals were initially assessed. Residues were collected from the scapula (F57S) and mandible (F57M) of burial F57 and from the femur (G336F) of burial G336 (**Figure 7.17**). A portion of associated orange material (F57R) was also collected from F57. In addition, images from the excavation of G336 showed basal grave deposits similar to those from within the coffin of the 'Spitalfields Lady'. Evaluation of these was, therefore, considered important and some of these materials were subsequently located and sampled. These comprised a portion of chalk rubble from the grave cut, samples of 'contaminated' fill from immediately above the lid of the lead-liner and deposits from the base of the liner (#9). Although the bulk of the materials from within the coffin had been wet-sieved (only artefacts/ecofacts were retained), fragments of mineral-replaced textiles and re-deposited lead, which appeared to have been untreated, remained extant. This work was made possible with the permission of Winchester Museums, facilitated by Helen Rees. Images of each sample with corresponding TICs and tabulated results are provided in **Appendix 6.4; Disc 1, File 6.4**.

Figure 7.17. Skeletal elements, Roman period, lead-lined coffin burials, Winchester, UK: a. mandible, F57, St Martin's Close; b. white 'tide mark', spinous process, lumbar vertebra, F57, St Martin's Close; c. shaft of left femur, G336, Eagle Hotel site (Author). Scale bars: 1 cm.

7.5.3 Results

7.5.3.1 Initial findings (residues from the skeletal remains)

Dark stains were impregnated into the skeletal elements of both individuals (G336; F57) with white material (plaster?) adhering to those of F57. Samples of these residues were minimally soluble in the organic solvent and contained low levels of lipids. Along with the orange fragments from F57, they were dominated by varying amounts of phthalate plasticisers, modern contaminants probably derived from the containers used to store the finds.

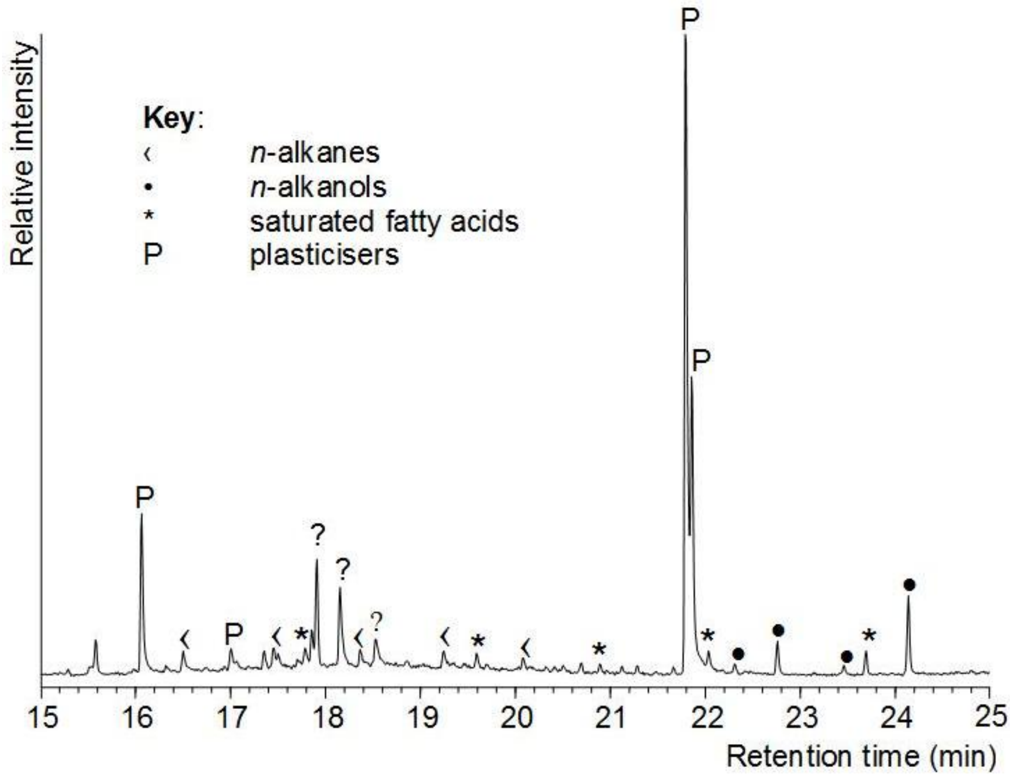


Figure 7.18. Partial TIC (15-25 min) residue, scapula, F57, St Martin's Close, Winchester, UK.

The compounds identified in the residue from the scapula (F57S) consisted of a limited range of *n*-alkanes (C_{18}-C_{25}), *n*-alkanols (C_{17}-C_{25}) and SFAs ($C_{12:0}$-$C_{20:0}$) (**Figure 7.18**). Likewise, the residue from the mandible (F57M) provided evidence for *n*-alkanes (C_{16}-C_{30}) and SFAs ($C_{14:0}$-$C_{22:0}$) with traces of $C_{18:1}$, sterols (cholesterol; brassicasterol?; stigmasterol; *β*-sitosterol) and 5α-cholestanol while the associated debris (F57R) contained *n*-alkanes (C_{17}-C_{25}), *n*-alkanols (C_{12}-C_{18}) and SFAs ($C_{14:0}$-$C_{18:0}$). The sample from the femur of G336 (G336F) from the Andover Road site also contained only low abundance *n*-alkanes (C_{17}-C_{22}) and *n*-alkanols (C_{14}-C_{22}) together with cholesterol and dehydrocholesterol. In this case, the sterols probably indicate relatively recent handling of the bone without gloves as 7-dehydrocholesterol is abundant in human skin (Wheatley and Reinertson 1958).

7.5.3.2 Control samples from G336 (outside the lead-liner)

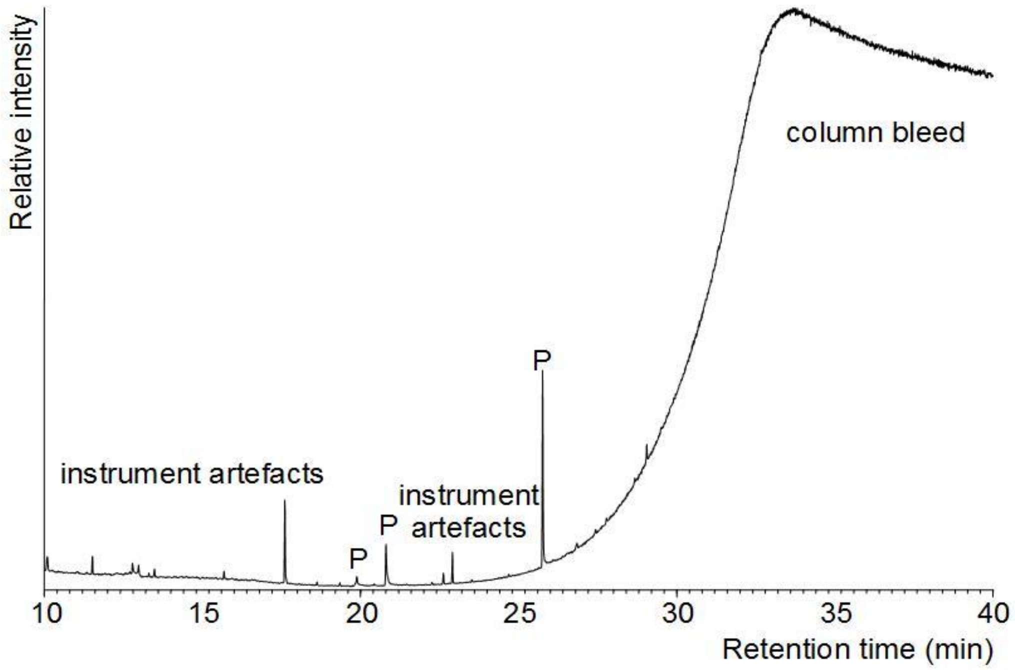


Figure 7.19. TIC control sample, fill external to the lead-liner, G336, Eagle Hotel site, Winchester, UK.

The sample of chalk rubble from the grave cut and sub-samples of the 'contaminated' fill from above the lid of the lead liner, which also appeared to largely consist of calcitic materials, were selected as controls indicative of the burial environment. The inorganic nature of these materials was confirmed by the near absence of lipid moieties, even those commonly indicative of soil organic matter (**Figure 7.19)**.

7.5.3.3 The grave deposits from G336

The grave deposits from the base of the coffin had been wet sieved upon excavation for the recovery of small finds and environmental evidence so little remained for analysis. Extant materials were predominantly inorganic in nature but some, such as the mineral-replaced textiles and fragments of lead with adhering residues, appeared to have been collected without subsequent treatment. Low levels of lipids belonging to a number of commonly observed classes (*n*-alkanes, *n*-alkanols and SFAs) were identified in the majority of these samples. Sterols and stanols of plant (campesterol; *β*-sitosterol; campestanol; stigmastanol) and animal (cholesterol; 5α-cholestanol) origin were also variously noted. This combination of input sources is typical of soil organic matter and/or the microbial degradation of intrinsic deposits (e.g. human remains, floral offerings, degraded textiles).

In addition, the highly persistent diterpenic end product, methyl dehydroabietate (MDHA) was observed in all of the samples with the exception of the flot (WN4). Of itself, this compound would not be considered significant. However, traces of diterpenoid resin acids (PM; SDPM) and dehydroabietic acid (DHA), the initial derivative of abietic acid, were also observed in the samples from Contexts 751 <3> and <4>, the mineral replaced textiles (WN13) and lead fragments (WN12) from the base of the coffin (**Figure 7.20; Table 7.10**).

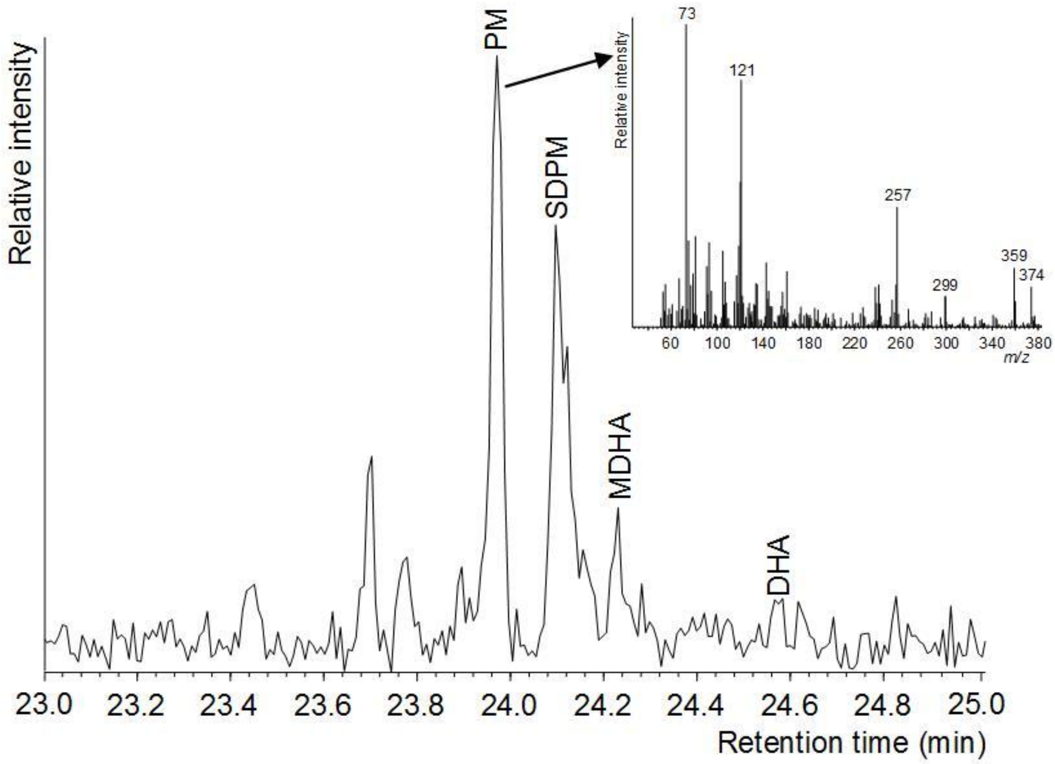


Figure 7.20. Partial XIC (*m/z* 121) grave deposits associated with lead fragments, WN12, lead-lined coffin burial (G336), Eagle Hotel site, Winchester, UK. Peak identifiers relate to **Table 7.10**. **Inset:** mass spectrum, pimaric acid (TMS derivative).

Although present in low abundance, these compounds are indicative of the presence of a Pinaceae resin (Colombini and Modugno 2009; Langenheim 2003: 37, 54-59). Comparison with modern Pinaceae products (**Appendix 3.1**) confirmed that the diterpenic compounds in the archaeological samples and reference materials displayed identical mass spectra. The survival and predominance of the pimarane skeleton resin acids in these trace level samples suggests that extensive, if any, thermal alteration prior to deposition is unlikely despite the presence of MDHA (**5.3.2; 8.5**).

Table 7.10. Identification of diterpenic compounds (TMS derivatives), grave deposits associated with lead fragments, G336, Winchester based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
PM	374	73	121, 133, 191, 207, 257, 299, 359	Pimaric acid
SDPM	374	121	73, 91, 143, 241, 257, 359	Sandaracopimaric acid
MDHA	314	239	55, 141, 239, 255, 299	Methyl dehydroabietate
DHA	372	239	73, 129, 143, 171/3, 185, 240, 255	Dehydroabietic acid

The solvent extract of the mineral-replaced textiles (WN13) also contained low levels of phenolic compounds (**Figure 7.21**). These volatile aromatics are present in the tissues of a range of botanical species (e.g. cinnamon/cassia) including balsamic resins extruded by members of the genera *Styrax* and *Liquidambar* (Modugno *et al.* 2006a; **5.5**). The presence of a number of pentacyclic triterpenic moieties in both WN13 and the sample associated with the lead fragments (WN12) is, therefore, highly significant. These were found to comprise compounds with olean-12-ene and urs-12-ene skeletons (based on their characteristic fragmentation patterns with ions at *m/z* 189, 203, 218, 279 and 320) which are of relatively widespread occurrence (Budzikiewicz *et al.* 1963; Modugno *et al.* 2006b). For example, the pentacyclic alcohols, α- and β-amyrin, are common components of angiosperms (flowering plants) although particularly abundant in resins produced by members of the Burseraceae (e.g. *Canarium, Bursera* and *Protium* spp.; Mathe *et al.* 2009; Mills and White 1977; Stacey *et al.* 2006). Based on their fragmentation patterns, the remainder of the triterpenoids present in these samples appeared to comprise oleanonic/ursonic (●) and oleanolic/ursolic (‡) isomers and a 3α/3β epimer of one of the latter (†) (**Figure 7.22; Table 7.11**). Secure identification was, however, hampered by the near identical mass spectra of these pairings.

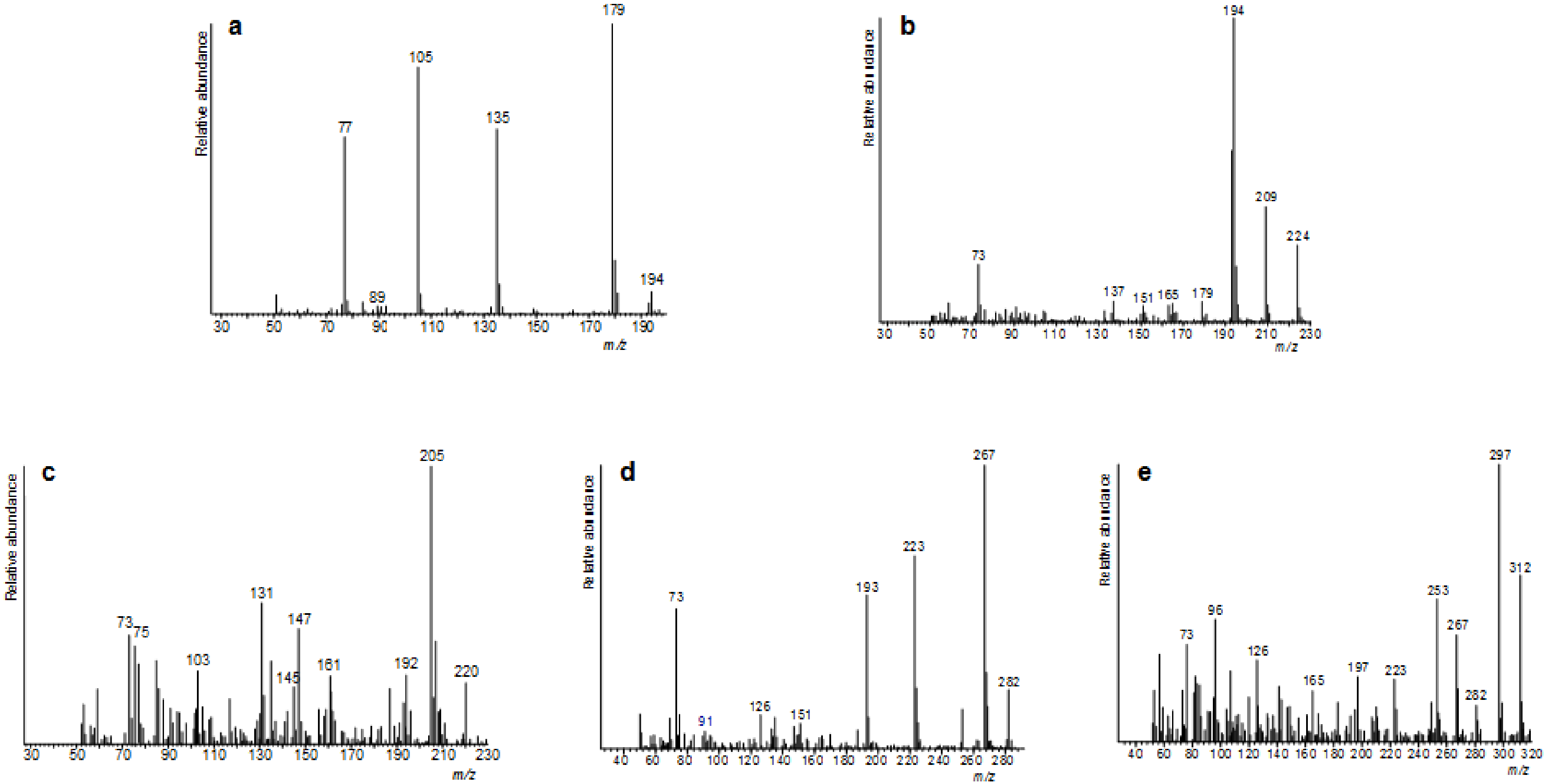


Figure 7.21. Phenolic compounds, solvent extract (WN13), mineral-replaced textiles, lead-lined coffin burial (G336), Eagle Hotel site, Winchester, UK ascribed as: a. benzoic acid; b. vanillin (4-hydroxy-3-methoxybenzaldehyde); c. cinnamic acid (3-phenylprop-2-enoic acid); d. 3-hydroxybenzoic acid; e. vanillic acid (4-hydroxy-3-methoxybenzoic acid) based on molecular ion ($M^{+}\bullet$), base peak (BP) and key fragment ions (TMS derivatives).

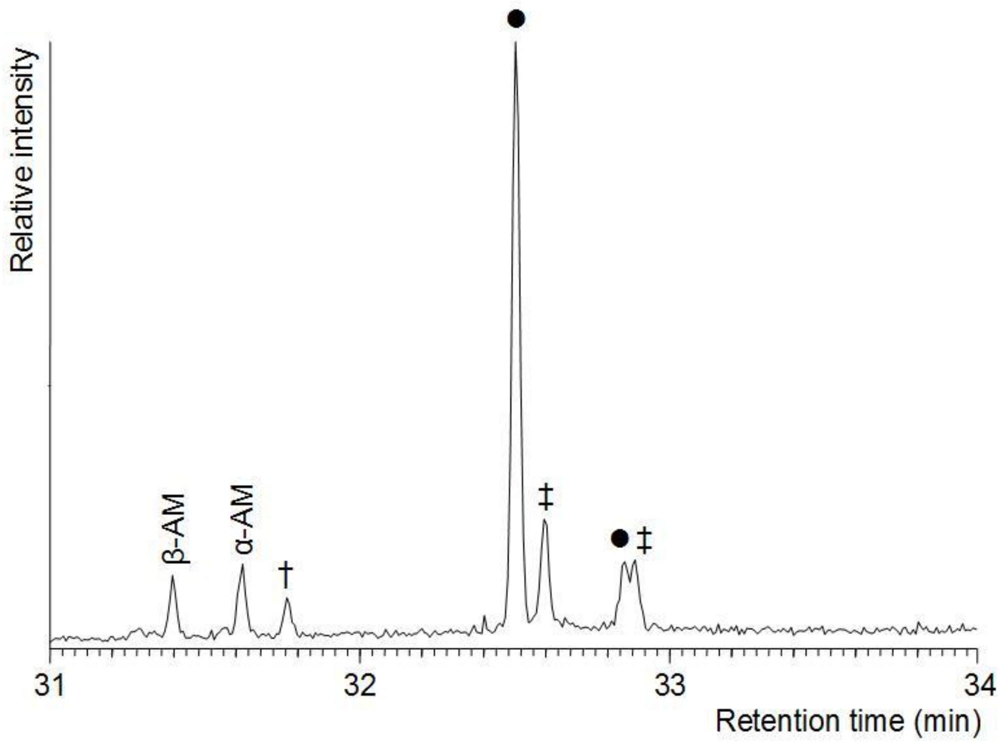


Figure 7.22. Partial XIC (*m/z* 203) mineral-replaced textile sample (WN13), lead-lined coffin burial (G336), Eagle Hotel site, Winchester, UK. Peak identifiers relate to **Table 7.11**.

Table 7.11. Provisional identification, triterpenic compounds (TMS derivatives) observed in samples (WN12; WN13), lead-lined coffin burial (G336), Eagle Hotel site, Winchester, UK.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
β-AM	498	218	483, 468, 408, 393, 311, 241, 203>189, 129	β-amyrin
α-AM	498	218	483, 468, 408, 393, 279, 257, 203=189, 135	α-amyrin
†	600	203	585, 510, 482, 320, 279, 189, 129, 73	3α/3β epimer of oleanolic or ursolic acid
●	526	203	511, 408, 393, 320, 307, 219, 189, 133, 119	**oleanonic** or ursonic acid
‡	600	203	585, 510, 482, 320, 279, 189, 129, 73	3α/3β epimer of **oleanolic** or ursolic acid
●	526	203	511, 408/9, 393, 320, 307, 219, 189, 133, 119	oleanonic or **ursonic** acid
‡	600	203	585, 510, 482, 320, 279, 189, 133, 73	3α/3β epimer of oleanolic or **ursolic** acid

A review of the literature with regards these tentatively identified compounds revealed that 3β-oleanolic acid has been documented in the tissues of over 1620 botanical species often in combination with its isomer, 3β-ursolic acid (Pollier and Goossens 2012). The 3α-epimers and related 3-oxo (=O at C-3) moieties (oleanonic and ursonic acid) appear to be of less frequent occurrence. Oleanonic acid is considered a key biomarker for *Pistacia* spp. resins but only in conjunction with moronic, *iso*masticadienonic and masticadienonic acid and/or their derivatives (Modugno *et al.* 2006b). Ursonic acid has been observed as a minor component in some dammar resins, exudates obtained from trees of the Dipterocarpaceae family native to SE Asia (Mills and Werner 1955), and, with 3β-ursolic acid and related compounds, in *Protium* spp. and *Bursera* spp. resins, predominantly native to the Americas (Stacey *et al.* 2006; Syamasunder *et al.* 1991).

Both 3α-*epi*oleanolic and oleanonic acid have, however, been reported in the balsamic exudate of *Liquidambar orientalis* (Huneck 1963) together with a number of unspecified isomers (Pastorova *et al.* 1998). Thus, as the original geographical distribution of many of these species rules them out as sources, the combination observed in the samples from Winchester most closely resembled the range of compounds (phenolics and triterpenoids) reported in balsamic exudates (**5.5**).

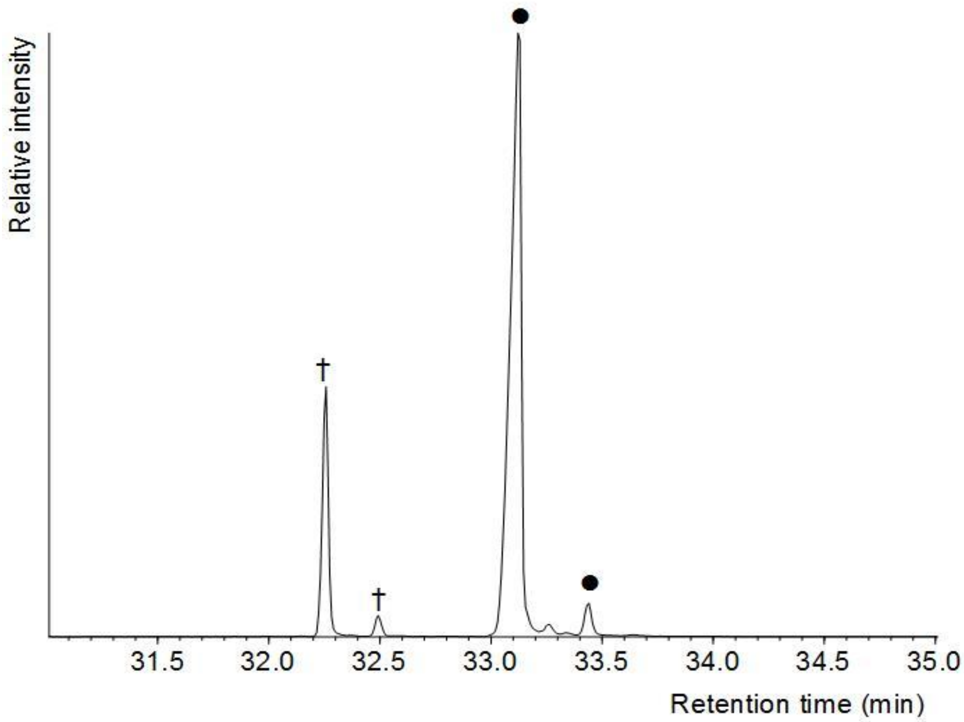


Figure 7.23. Partial XIC (*m/z* 203) triterpenic compounds, modern reference sample, *Liquidambar orientalis,* raw extract (STOSLR; Soma Luna), Turkey. Peak identifiers relate to **Table 7.12**.

Table 7.12. Provisional identification, triterpenic compounds (TMS derivatives) present in the modern *Liquidambar orientalis* raw (STOSLR) and bark extracts (STOSLB) analysed.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
†	600	203	585, 510, 482, 320, 279, 189, 129	3α/3β epimer of **oleanolic** or ursolic acid
†	600	203	585, 510, 482, 320, 279, 189, 129	3α/3β epimer of oleanolic or **ursolic** acid
●	526	203	511, 408, 393, 320, 307, 219, 189, 133	**oleanonic** or ursonic acid
●	526	203	511, 408/9, 393, 320, 307, 219, 189, 133	oleanonic or **ursonic** acid

This survey also revealed that limited research had been undertaken with regards the chemical composition of these balsamic resins, particularly *S.*

officinalis (Serpico 1996: 58-60). Moreover, lack of specification and variable identification of the epimers present and even differences in interpretation of the elution order of these compounds was noted in publications concerning *L. orientalis* resins (Hovaneissian *et al.* 2008; Modugno *et al.* 2006a; Pastorova *et al.* 1997, 1998), although the weight of evidence indicated that oleanane-skeleton compounds elute before their corresponding ursane-skeleton isomers (Burnouf-Radosevich *et al.* 1985; embolded **Tables 7.11, 7.12**). Of the modern exudates analysed, those from *L. orientalis* displayed some similarities (**Figure 7.23; Table 7.12**) to the data obtained from the archaeological samples but, without precise identification of each compound, questions remained.

7.5.4 Characterisation of balsamic resins

7.5.4.1 Synthesis of key triterpenoids

In order to confirm the identity of each of the compounds present in *L. orientalis* resins, 3β-oleanolic and 3β-ursolic acid standards were purchased from Tokyo Chemical Industry (TCI) UK Ltd. Industry standards of their corresponding 3α-epimers and 3-oxo derivatives were not available. Thus, the 3β-epimers were oxidised using Jones reagent to produce the C-3 position ketones (oleanonic and ursonic acid) followed by reduction with the bulky reagent *L*-selectride® to generate the corresponding 3α-epimers (**Figure 7.24)**, under the direction of Dr William Martin, Lecturer in Organic Chemistry, University of Bradford.

Completion of each reaction was monitored using thin layer chromatography (TLC) with the purity and identity of the synthesised compounds confirmed using ^{1}H NMR. The 3β-epimers in which the –OH substituent at C-3 is equatorial (i.e. parallel to the plane of the A ring) displayed a doublet at around 2.8 ppm for oleanolic acid and 2.2 ppm for ursolic acid. This was replaced by a doublet of multiplets between 2.3-2.7 ppm in the 3-oxo derivative due to the deshielded ketone at C-3 with reappearance of the doublet with a small upfield shift (c. 2.7 and 2.1 ppm) in the 3α-epimers where the –OH substituent is axial (i.e. perpendicular to the A ring). Since axial substituents create steric strain in ring structures, the 3β-epimer is the more stable conformation which indicates that the 3α-epimer might be more readily degraded or modified in a burial environment although this has yet to be tested.

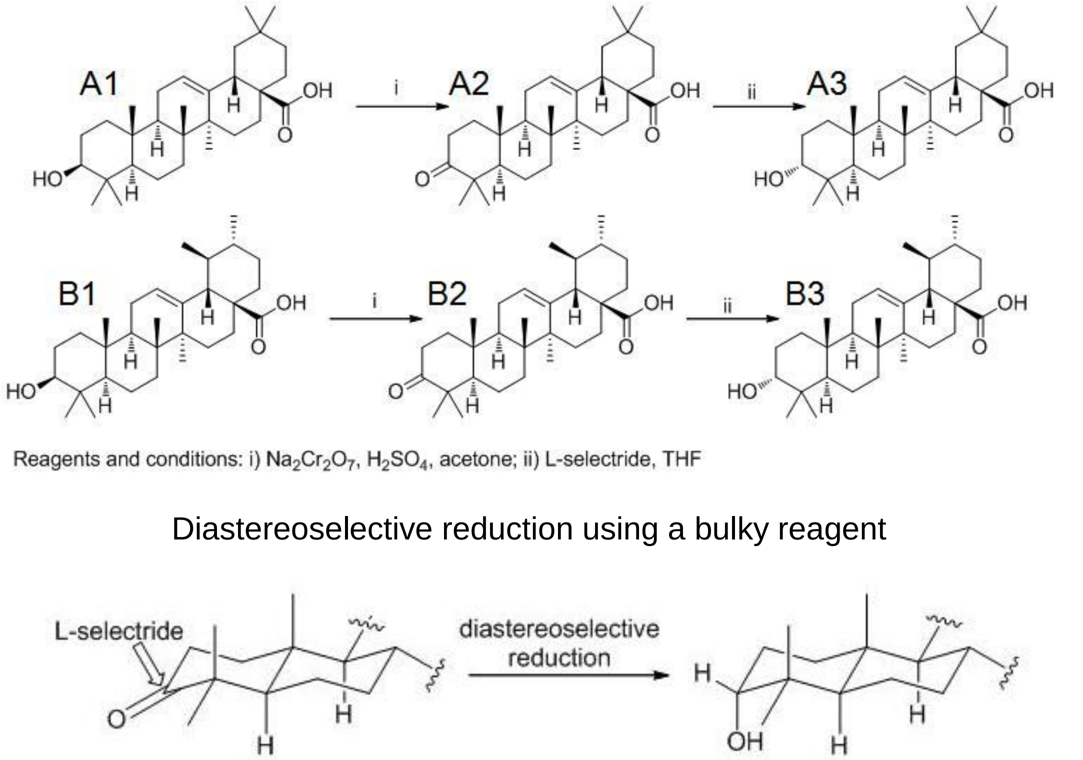


Figure 7.24. The synthetic method, showing the structures of the purchased and synthesised standards (A1. 3β-oleanolic acid; A2: oleanonic acid; A3. 3α-oleanolic acid; B1. 3β-ursolic acid; B2: ursonic acid; B3. 3α-ursolic acid): i. oxidation of the 3β-epimeric alcohols (A1; B1) using Jones reagent ($Na_2Cr_2O_7$, H_2SO_4); ii. diastereoselective reduction of the ketones (A2; B2) using *L*-selectride® to produce the 3α-epimeric alcohols (A3; B3) by the pathway shown.

Further evaluation of the behaviour of these epimeric pairs under positive mode APCI conditions was undertaken, facilitated by Dr Richard Gallagher (AstraZeneca). Initial findings revealed a more facile loss of a water molecule, the principal primary fragment of the protonated acids, from the 3β-epimer (**Figure 7.25**). This occurred as a result of divergent rearrangements due to the different environments experienced by the hydroxyl group at C-3 (i.e. at the migration terminus). A mechanism for this reaction was proposed by Dr Martin (**Figure 7.26**). The subsequent synthesis of related epimeric pairs (undertaken by Chloe Townley; **Appendix 7.6**) demonstrated a systematic difference in fragmentation (APCI +ive mode) dependent on the stereochemistry of the hydroxyl group at C-3 and has provided a novel approach for distinguishing between steroidal epimers using APCI-LC-MS (Townley *et al.* 2015).

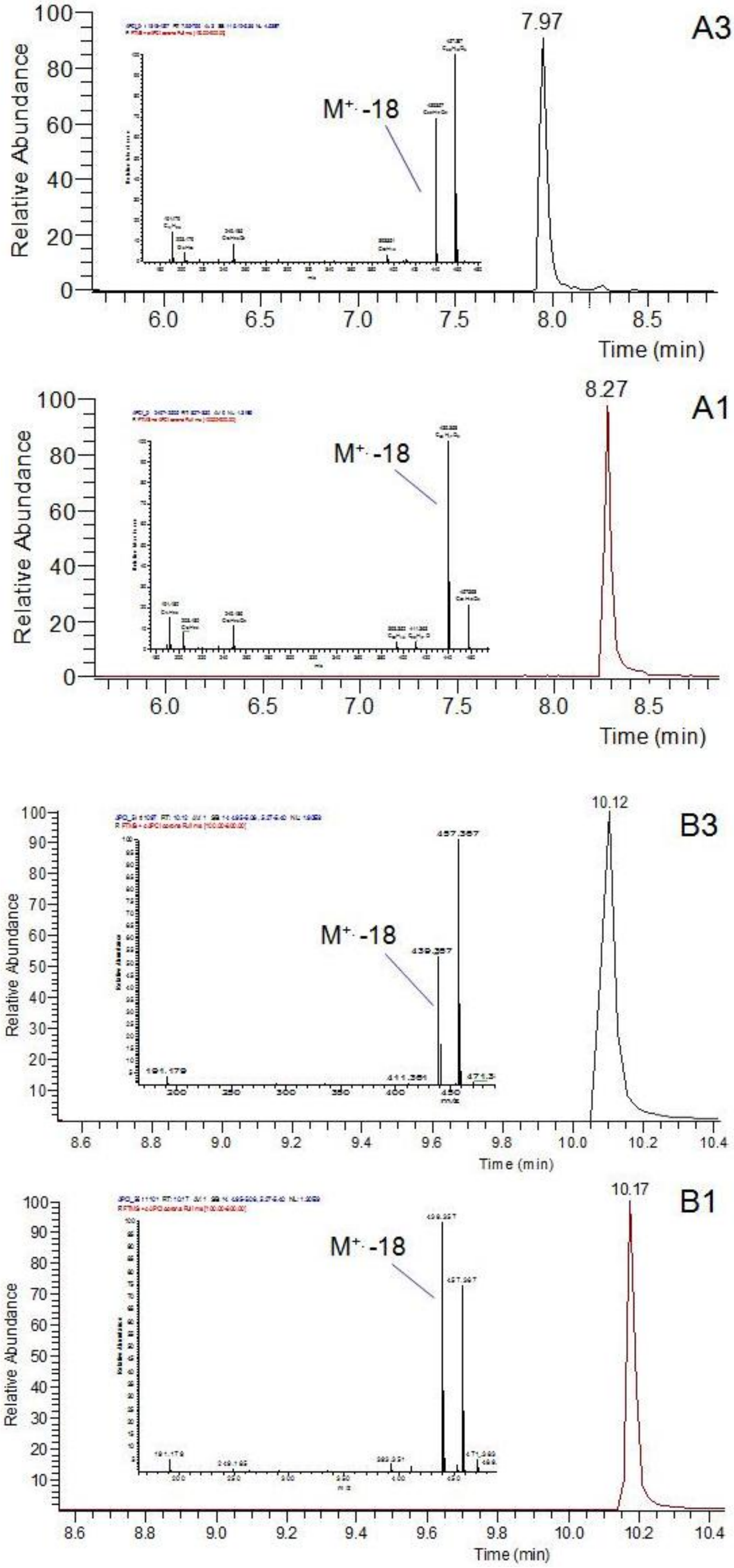


Figure 7.25. APCI +ive ion, Orbitrap MS, accurate mass chromatograms of the epimeric alcohols discussed in the text showing the more facile loss of a water molecule from the 3β-epimer: A1. 3β-oleanolic acid; A3. 3α-oleanolic acid; B1. 3β-ursolic acid; B3. 3α-ursolic acid.

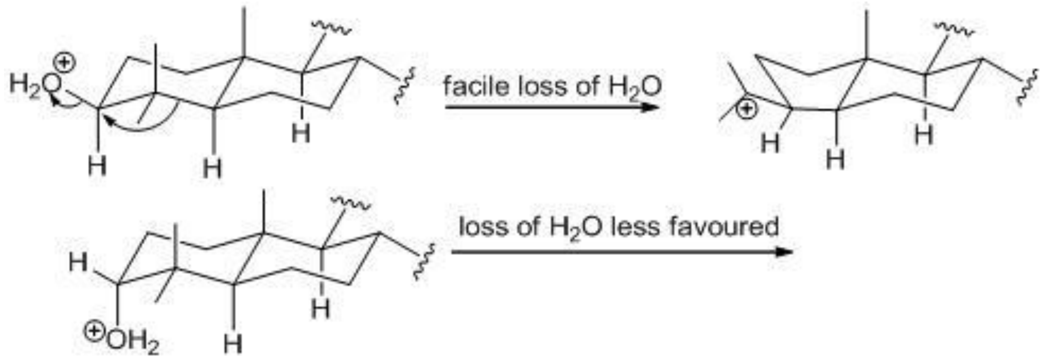
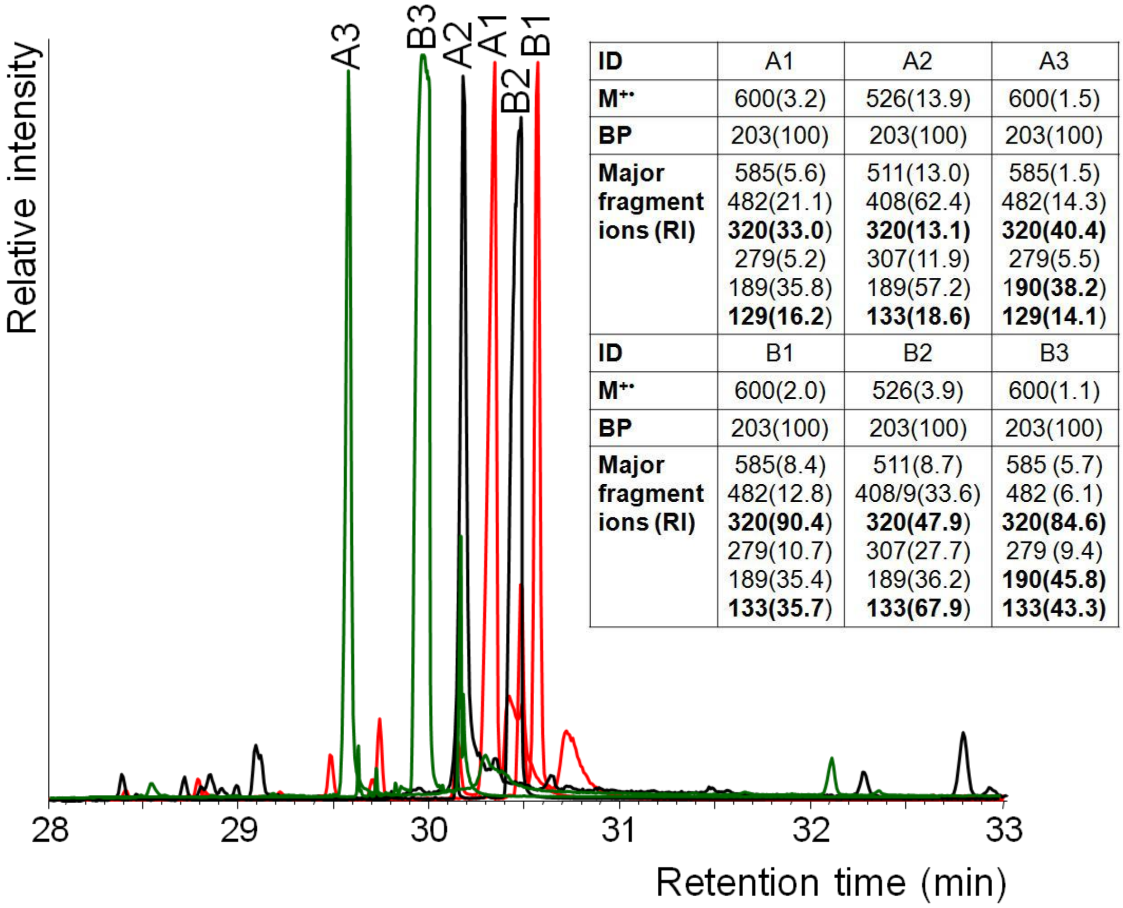


Figure 7.26. Proposed mechanism for the more facile loss of water when the hydroxyl group is in the equatorial position, i.e. from the 3β-epimer.

7.5.4.2 Characterisation using GC-MS

The elution order and mass spectral fragmentation patterns of the synthesised compounds were then established using GC-MS (**Figure 7.27**).



ID	A1	A2	A3
M$^{+\bullet}$	600(3.2)	526(13.9)	600(1.5)
BP	203(100)	203(100)	203(100)
Major fragment ions (RI)	585(5.6) 482(21.1) **320(33.0)** 279(5.2) 189(35.8) **129(16.2)**	511(13.0) 408(62.4) **320(13.1)** 307(11.9) 189(57.2) **133(18.6)**	585(1.5) 482(14.3) **320(40.4)** 279(5.5) **190(38.2)** **129(14.1)**
ID	B1	B2	B3
M$^{+\bullet}$	600(2.0)	526(3.9)	600(1.1)
BP	203(100)	203(100)	203(100)
Major fragment ions (RI)	585(8.4) 482(12.8) **320(90.4)** 279(10.7) 189(35.4) **133(35.7)**	511(8.7) 408/9(33.6) **320(47.9)** 307(27.7) 189(36.2) **133(67.9)**	585 (5.7) 482 (6.1) **320(84.6)** 279 (9.4) **190(45.8)** **133(43.3)**

Figure 7.27. Partial XIC (*m/z* 203) showing the relationship between the triterpenoids of interest (TMS derivatives): A1. 3β-oleanolic acid; A2: oleanonic acid; A3. 3α-oleanolic acid; B1. 3β-ursolic acid; B2: ursonic acid; B3. 3α-ursolic acid. **Inset:** mass and relative intensity of ions (% in respect of BP).

This analysis revealed key differences with the common fragment ion at *m/z* 320 considerably more abundant in all of the ursane-skeleton moieties (TMS derivatives). Those with an hydroxyl group at C-3 were additionally distinguished by a fragment ion at *m/z* 133 (ursanes) and *m/z* 129 (oleananes). Although these characteristic ions were present in both the 3α- and 3β-epimers, differentiation between the epimeric pairs was readily achievable due to the considerably shorter retention time of the former. Thus, the 3α-epimer was found to elute 0.5-0.6 minutes prior to the corresponding ketone with the 3β-epimer appearing approximately 0.1-0.2 minutes after the corresponding ketone.

Such comparisons may, however, not always be possible (i.e. where no ketone and/or only one epimer is present), so further differences in fragmentation patterns were evaluated. This revealed a fragment ion at *m/z* 189 in the 3β-epimer with a shift to *m/z* 190 in the 3α-epimer. Likewise, the greater relative abundance of the molecular ion, M-15 and M-188 fragments in the 3β-epimer appears to be a diagnostic feature. These observations match well with the data presented by Burnouf-Radosevich *et al.* (1985).

7.5.4.3 Triterpenoids in modern balsamic exudates

These observations were used to enable secure identification of the triterpenoids present in modern balsamic exudates. The Turkish *Liquidambar orientalis* raw extract (STOSLR) and bark (STOSLB) from Soma Luna and the extract (STOBB) from Bristol Botanicals Ltd. were all found to contain the 3α-epimers of oleanolic (EOLA) and ursolic (EULA) acid alongside oleanonic (ONA) acid and ursonic (UNA) acid with a greater abundance of oleanane-skeleton moieties and no evidence of the 3β-epimers (**Figure 7.28; Table 7.13**). Related terpenic compounds were also observed together with bound and free phenolic compounds, dominated by cinnamyl cinnamate (**5.5**) and traces of 3-*epi*-β-amyrin and 3-*epi*-α-amyrin (base peak 218, eluting c. three minutes prior to ONA, Soma Luna samples). The purified extract (STOSLP), however, contained only traces of free phenolics demonstrating the significant impact of processing on chemical composition (**Figure 7.29**).

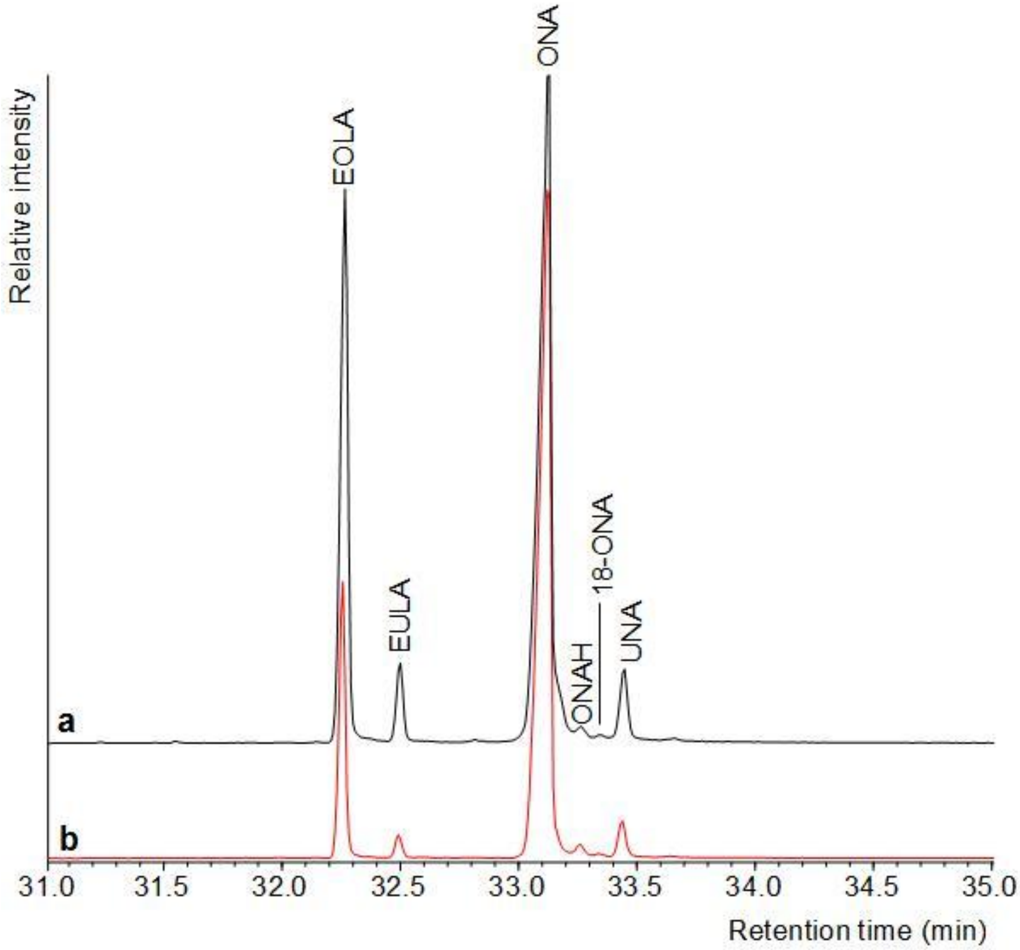


Figure 7.28. Partial XIC (*m/z* 203) secure identification of the triterpenic compounds (TMS derivatives) in modern *L. orientalis* balsams from Turkey: a. raw extract (STOSLR), INC151, Soma Luna; b. extract (STOBB), GAW017, Bristol Botanicals Ltd.

Table 7.13. Identification of triterpenic compounds (TMS derivatives), modern *L. orientalis* extracts based on elution order, molecular ion (M^{+}•), base peak (BP) and key fragment ions: *experimentally determined; **identified through comparison with the literature (Papageorgiou *et al.* 1997; Stern *et al.* 2003).

Peak	M^{+}•	BP	Key fragment ions	Name of compound
EOLA	600	203	585, 510, 482, 320, 279, 189, 129, 73	3α-epimer of oleanolic acid*
EULA	600	203	585, 510, 482, 320, 279, 189, 129, 73	3α-epimer of ursolic acid*
ONA	526	203	511, 408, 393, 320, 307, 219, 189, 133	oleanonic acid*
ONAH	438	203	408/9, 320, 232, 189, 175, 133, 119. 105	oleanonic aldehyde**
18-ONA	526	203	511, 408, 393, 320, 307, 219, 189, 133	18αH-oleanonic acid**
UNA	526	203	511, 408/9, 393, 320, 307, 219, 189, 133	ursonic acid*

Comparison with published data shows that, in all instances, two abundant triterpenoids and a number of minor peaks are present in *L. orientalis* exudates with oleanonic acid the dominant compound (cf. Modugno *et al.* 2006a; Pastorova *et al.* 1998). The second significant peak can now clearly be identified as the 3α-epimer of oleanolic acid based on elution order and the findings obtained from the extracts analysed for this project. It should be noted, however, that the fragment ions and their relative intensities reported by Pastorova *et al.* (1998; **Figure 7.30**) appear to reflect those observed both here and by Burnouf-Radosevich *et al.* (1985) in ursane-skeleton compounds (*m/z* 190, an abundance of *m/z* 320) rather than oleananes.

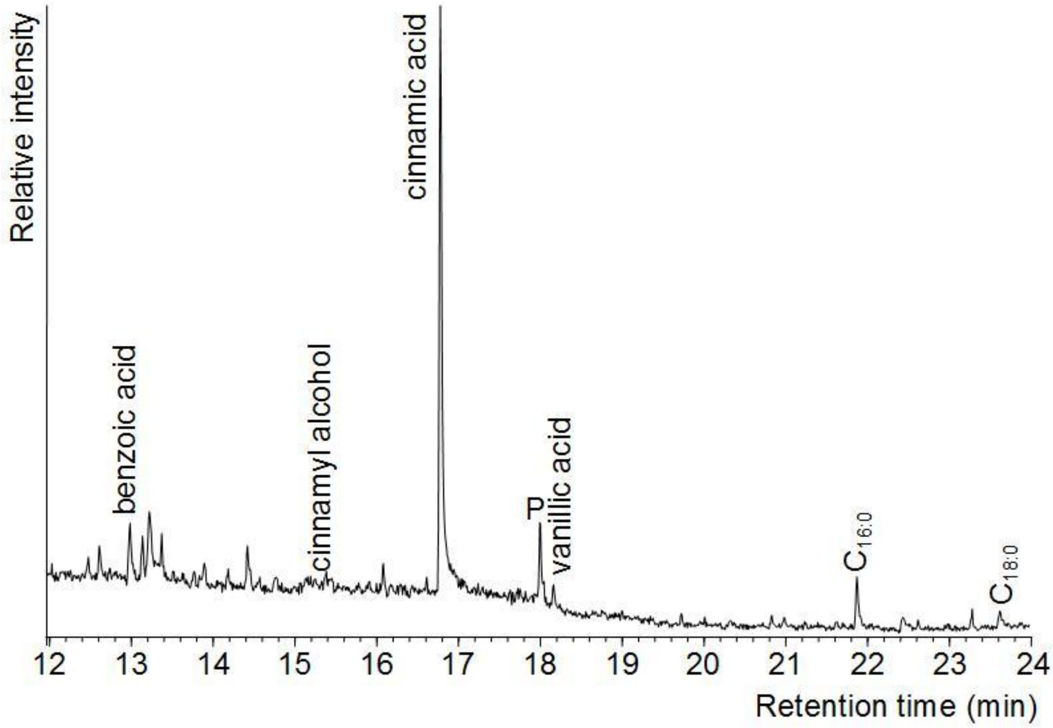


Figure 7.29. Partial TIC (12-24 min) phenolic compounds and SFAs (TMS derivatives) identified in the modern *L. orientalis* purified extract (STOSLP; INC084, Soma Luna), Turkey.

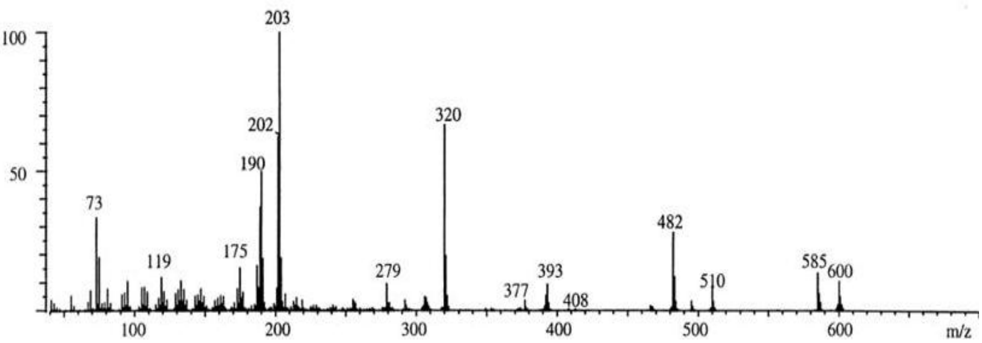


Figure 7.30. Mass spectrum of compound identified as 3-epi-oleanolic acid (TMS derivative), *L. orientalis exudate* (storax), Turkey (Figure from Pastorova *et al.* 1998: Figure 2B, 1384).

As exudates from *Styrax officinalis* were not able to be obtained, twigs from a sapling grown in the UK (STYBNT) were pulverised and solvent extracted. A range of lipid species common in vegetative matter were identified (*n*-alkanes, *n*-alkanols, SFAs, phytosterols, phytostanols) together with an array of phenolics including benzoic acid and its derivatives although cinnamates were not observed. Evidence for triterpenic compounds with oleanane and ursane skeletons was obtained although most of these could only be tentatively identified (**Figure 7.31; Table 7.14**). No resin acids were present.

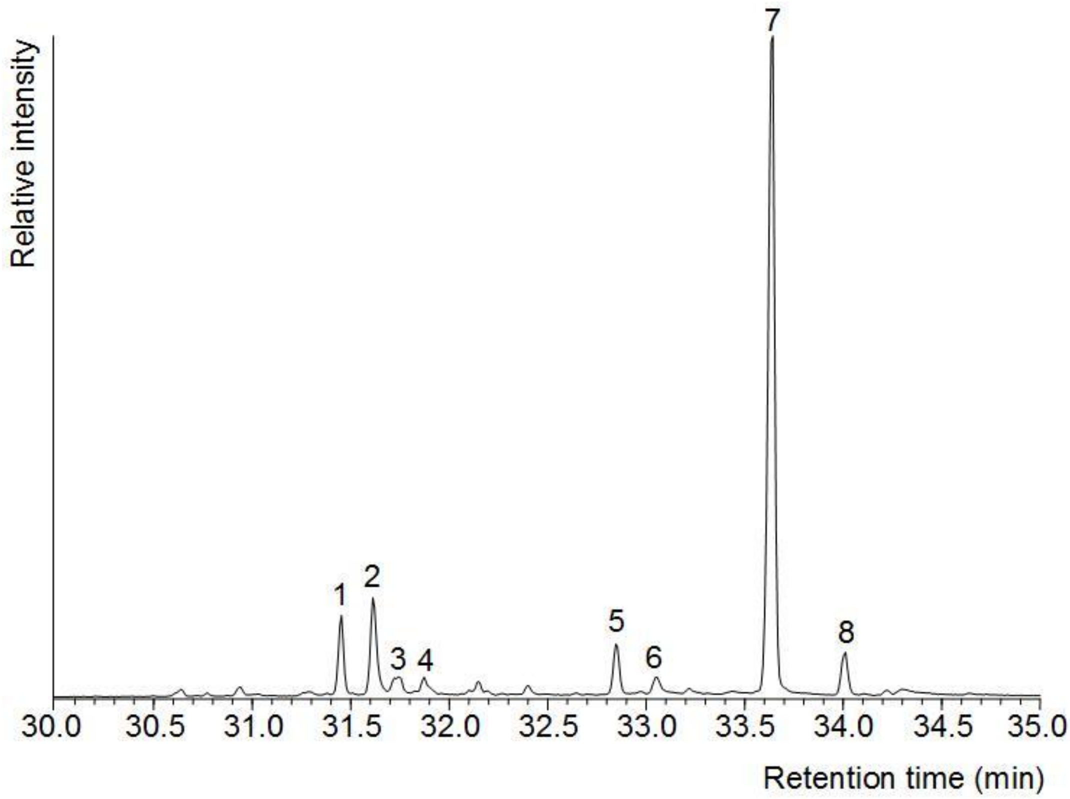


Figure 7.31. Partial XIC (*m/z* 203) triterpenic compounds present in the solvent extract of modern *Styrax officinalis* twigs (STYBNT; sapling from Burncoose Nurseries, UK).

Table 7.14. Tentative identification, triterpenic compounds (TMS derivatives), solvent extract, modern pulverised *S. officinalis* twigs (STYBNT) based on comparison with the literature (Assimopoulou and Papageorgiou 2005a, 2005b; Papageorgiou *et al.* 1997; Stern *et al.* 2003).

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
1	424	218	409, 269, 257, 231, 203, 189, 137, 121	β-amyrenone
2	424	218	411, 281, 257, 231, 203, 189, 175, 135	?α-amyrenone
3	426	218	411, 393, 257, 229, 203, 189, 135, 121	?olean-12-ene derivative
4	468	189	453, 393, 257, 218, 203, 175, 135, 121	?olean-18-ene derivative
5	438	203	409, 232, 189>175, 133, 119	oleanonic aldehyde
6	438	203	409, 232, 189>=175, 131, 119	ursonic aldehyde
7	482	203	467, 393, 281, 253, 232, 189, 175, 133	?11-oxo-β-amyrin acetate
8	482	203	467, 393, 281, 253, 232, 189, 175 133	?11-oxo-α-amyrin acetate

7.5.4.4 Triterpenoids in the archaeological samples

The terpenic compounds present in the samples from burial G336, Eagle Hotel site, Winchester were, however, able to be accurately identified as a result of this research. In addition to the pentacyclic alcohols, β- and α-amyrin, these comprised a distinctive suite of resin acids (**Figure 7.32; Table 7.15**).

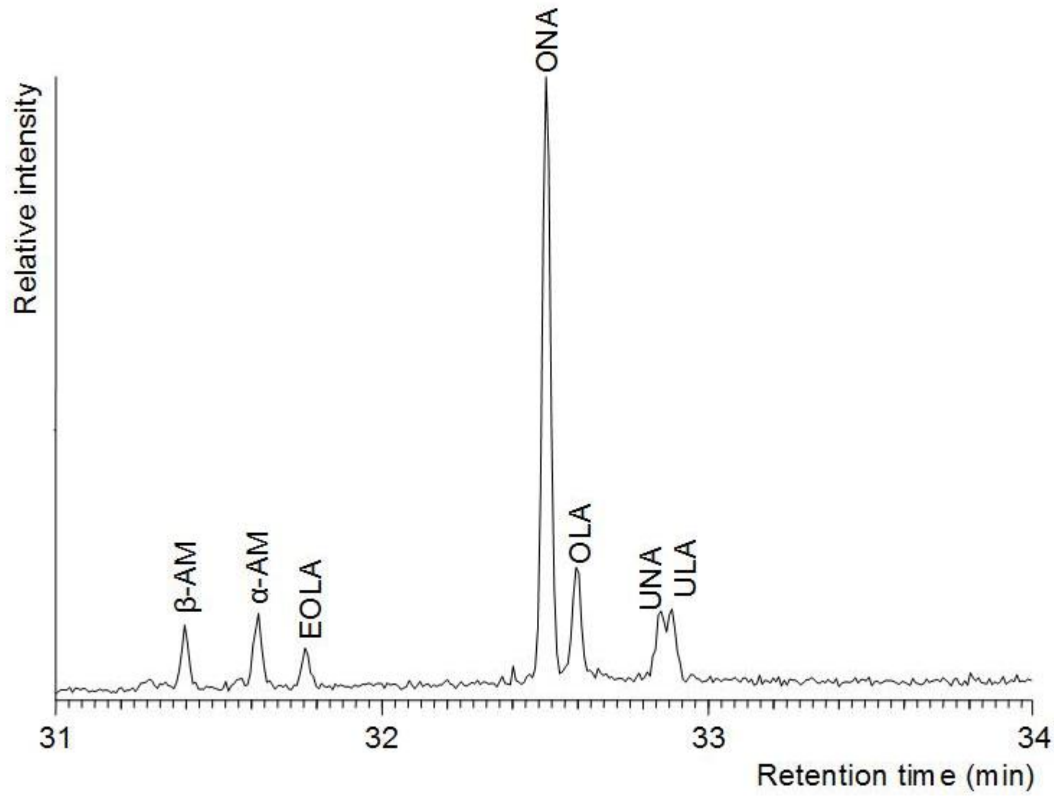


Figure 7.32. Partial XIC (*m/z* 203) terpenic compounds present in the mineral-replaced textile sample (WN13), lead-lined coffin (G336), Eagle Hotel, Winchester, UK. Peak identifiers relate to **Table 7.15**.

Table 7.15. Definitive identification of triterpenic compounds (TMS derivatives), archaeological samples, lead-lined coffin (G336), Eagle Hotel site, Winchester, UK based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
β-AM	498	218	483, 468, 408, 393, 311, 241, 203>189, 161, 129	β-amyrin
α-AM	498	218	483, 468, 408, 393, 279, 257, 203=189, 175, 135	α-amyrin
EOLA	600	203	585, 510, 482, 320, 279, 189, 129, 73	3α-*epi*oleanolic acid
ONA	526	203	511, 408, 393, 320, 307, 219, 189, 133, 119	oleanonic acid
OLA	600	203	585, 510, 482, 320, 279, 189, 129, 73	3β-oleanolic acid
UNA	526	203	511, 408/9, 393, 320, 307, 219, 189, 133, 119	ursonic acid
ULA	600	203	585, 510, 482, 320, 279, 189, 133, 73	3β-ursolic acid

Despite this positive result, the sequence of compounds in the Eagle Hotel samples did not correspond to those observed in the modern balsamic extracts, with that from *S. officinalis* significantly different. Indeed, this combination was not found in any of the triterpenoid-containing reference resins and gum-resins analysed as part of this project (**Table 6.1**; **Appendix 3.2-3.4**). It also seems not to have been reported in the phytochemical literature (at least in the many publications reviewed). Thus, although most similar to the suite of resin acids identified *L. orientalis* extracts, to put it simply, the 'wrong' epimers (i.e. predominantly 3β rather than 3α, including the pentacyclic alcohols) were present in the archaeological materials.

7.5.5 Summary and interpretation of findings

The residues associated with the skeletal remains of F57, St Martin's Close, Winnall and G336, Eagle Hotel, Winchester did not contain lipids of archaeological origin. The control samples selected from the fill around the coffin of the latter showed that the external burial environment was characterised by very low levels of ubiquitous organic compounds with no phenolic or terpenic contribution. Results obtained from analysis of various materials from within the lead-lined coffin can, therefore, be considered of archaeological relevance. The range and relative abundance of the *n*-alkanes and *n*-alkanols indicated that these deposits predominantly derived from degraded higher plant matter with some microbial contribution. The saturated carboxylic acids and combination of sterols and stanols present indicated the presence of both decomposed plant and animal tissues.

With regards to the terpenic compounds identified, methyl dehydroabietate (MDHA) was present in all of the samples. This neutral derivative has been shown to be an highly resistant end product of the degradation pathways of the abietane-skeleton resin acids found in Pinaceae products. As such, it is frequently observed in soil organic matter across the northern hemisphere although it is also produced by the heating of resinous Pinaceae woods (Hjulström *et al.* 2006). In this instance, the presence and relative abundance of pimarane-based resin acids (PM; SDPM) together with traces of dehydroabietic acid (DHA), the initial dehydration product of abietic acid, in four of the samples suggests that MDHA simply represents the most persistent indicator for the inclusion of a Pinaceae resin in this burial.

The survival of triterpenoid resin acids and LMM phenolic compounds, which were most clearly observed in the extract from the mineral-replaced textiles (WN13), supports this contention (i.e. an absence of heating) and provides evidence for the use of a least one other resinous substance. The current lack of a chemical match for this combination of compounds, however, gives rise to a number of hypotheses. These are that:

1. a mixture of substances, perhaps in the form of an unguent, was present with the different lipid species (phenolics, oleananes, ursanes) derived from different sources;
2. a single balsamic triterpenoid resin was used which, due to the confounding effects of natural variability, differential harvesting methods, or a decline in the exploitation of certain species, has not yet been identified;
3. the species from which the exudate was obtained has subsequently become extinct;
4. taphonomic conditions in the burial environment have altered the chemical composition of the resin in an unexpected manner.

With regards to *Hypothesis 1*, triterpenic compounds are present in the tissues of many plants and have been extracted/utilised for millennia (Howes 1950; Serpico 2000). For example, oil-producing plants (e.g. *Olea europaea,* the source of olive oil) and many aromatic herbs contain either oleanolic or ursolic acid or both, alongside related compounds (e.g. Bianchi *et al.* 1994; Liu 1995). Thus, the absence of a match with the reference materials may be due to the application of a mixture of substances, perhaps in the form of a scented unguent or balm. Where such mixtures have previously been identified in the archaeological record, the presence of an animal fat or plant oil (indicated by an abundance of SFAs, MUFAs and related compounds) as a major ingredient has provided supporting evidence (e.g. Buckley and Evershed 2001; Charrié-Duhaut *et al.* 2007; Colombini *et al.* 2009; Ribechini *et al.* 2009). This does not appear to be the case with G336 although confirmation cannot be obtained due to the limitations of the extant samples.

It is interesting to note, therefore, that a similar combination of compounds to those recovered from the samples from Winchester has been observed in a number of sarcophagus burials from St Maximin's, Trier, a glass vessel from a sarcophagus burial below St Matthias', Trier, samples from the tower tomb of Atenatan, Palmyra and textile fragments from Grave 530, Poundbury, Dorchester, Dorset, UK (Reifarth 2013: 96-99, 494, 507-508; **Figure 7.33; Table 7.16**, accessed courtesy of Carl Heron and Nicole Reifarth). In these examples, it was originally thought that a *Boswellia* spp. (interpretation of FTIR spectra) or *Pistacia* spp. (oleanonic acid revealed by GC-MS) exudate might be present. Nonetheless, evidence of phenolic compounds including cinnamates pointed to the possibility of a balsam (Heron 2011, pers. comm., November). Further work, based on the research detailed above, indicates that the latter interpretation is more likely. These findings appear to support the suggestion that the compounds in the samples from G336 are representative of a single resin with differences in the data primarily due to differential conditions within the burial environment (e.g. the production of *O*-acetyl derivatives in an oxidising environment).

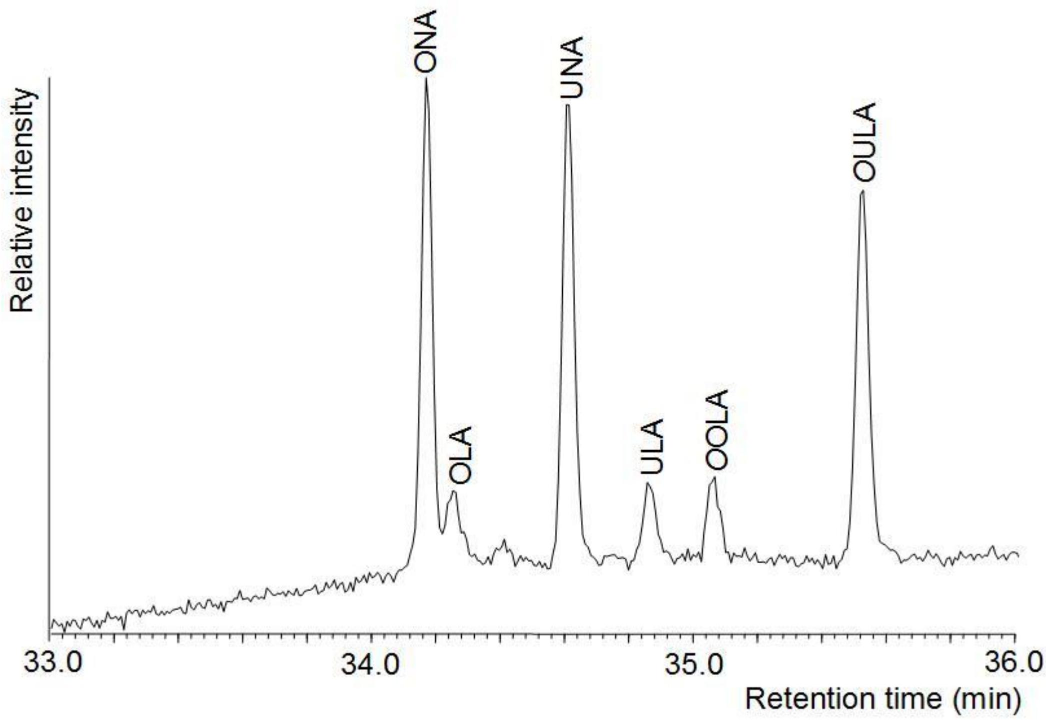


Figure 7.33. Partial XIC (*m/z* 203) solvent extract, headband, burial 530, Poundbury, Dorchester, UK. Peak identifiers relate to **Table 7.16**.

Table 7.16. Identification of triterpenoids (TMS derivatives), sample from the headband, Grave 530, Poundbury, Dorchester, Dorset, UK.

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
ONA	526	203	511, 408, 393, 320, 307, 219, 189, 133, 119	oleanonic acid
OLA	600	203	585, 510, 482, 320, 279, 189, 129, 73	3β-oleanolic acid
UNA	526	203	511, 408/9, 393, 320, 307, 219, 189, 133, 119	ursonic acid
ULA	600	203	585, 510, 482, 320, 279, 189, 133, 73	3β-ursolic acid
*O*OLA	570	203	555, 510, 452, 320, 189, 119, 73	*O*-acetyl-oleanolic acid
*O*ULA	570	203	555, 510, 452, 320, 307, 189, 133, 73	*O*-acetyl-ursolic acid

If, as stated in *Hypothesis 2*, these compounds do derive from a single botanical source, the traces of α- and β-amyrin are indicative of an angiosperm with the presence of both oleanane and ursane-skeleton triterpenoids significantly reducing the possibilities and eliminating many substances mentioned in ancient texts (e.g. labdanum, ammoniacum, galbanum, opopanax; **5.2; 5.3.2**). *Pistacia* spp. resins can also be ruled out since the presence of ursane-based compounds and absence of moronic acid and noroleanane derivatives does not match the composition even of aged or thermally altered samples (**5.3.3; Appendix 3.2;** Stern *et al.* 2003). Likewise, the boswellic acids and their degradation products, characteristic of frankincense, were not observed (**5.4; Appendix 3.3**).

Other members of the Burseraceae, such as elemi, are a possibility, although the pentacyclic alcohols tend to dominate in such resins (Mills and White 1999: 108; Stacey *et al.* 2006). In addition, the LMM phenolics suggest a balsam, with those known to be available in the Roman Empire restricted to *L. orientalis* or *S. officinalis*. As demonstrated above, neither of these provides an exact match although more work is required as regards the latter including the boiling and pressing of the bark to procure an extract as detailed by Guenther (1943). Unfortunately, the young tree purchased with this in mind (from which twigs were obtained) did not thrive, or indeed survive, in northern England and resinous extracts could not be obtained (despite extensive enquiries and assistance from researchers in Israel).

There is also the possibility that environmental change (resulting in an inability to produce exudates today), the exploitation in antiquity of trees not currently considered viable resin producers or the loss of the art of extraction from more challenging species (e.g. *S. officinalis*) may continue to confound identification. Indeed, the species utilised may even have become extinct (*Hypothesis 3*). To illustrate this problem, there is one attested example relating to a fennel-like species known as *silphium* (or *lasaron*) which grew wild along the north coast of Africa (mainly in Libya) (Gemmill 1966; Langenheim 2003: 413-417). The exudate of this plant was highly prized and widely used in antiquity as incense, in perfumes, for medicinal purposes and as a seasoning (*Theophrastus 6.3* nd). Indeed, it was of such economic significance in the region that it appeared on the coinage of Cyrene (Tatman

2001). Nonetheless, according to Pliny the Elder, by the 1st century AD, "*only a single stock has been found...within our memory, which was sent to the emperor Nero"* (*NH 19.15:29* nd) with silphium seemingly extinct by the early 3rd century AD (Gemmill 1966). It has been suggested that this plant may have belonged to the genus *Ferula* (Umbelliferae/Apiaceae) and so its extract is likely to have contained a range of phenolic and LMM terpenic compounds (e.g. ferulic acid, umbelliferone, mono- and sesquiterpenes; Langenheim 2003: 413-417). Thus, on the basis of the reported chemistry of extant *Ferula* spp. it is an unlikely candidate for the mystery balsamic.

The final option is that taphonomic processes have obscured the relationship between the resin used and the modern reference materials analysed (*Hypothesis 4*). This is a strong possibility since the microenvironment within these lead-lined coffin or stone sarcophagus burials confined the products of degradation (human remains, textiles, associated offerings, grave goods, inner surfaces of the burial containers) and often included calcareous materials (bone, plaster, limestone, ingress from surrounding geology). Although these conditions did not, in general, favour long-term skeletal preservation, they clearly facilitated the preservation of hair and the formation of mineral-replaced textiles due to substitution by metal ions. Could this specific environment have resulted in the natural conversion of the less stable 3α-oleanolic and 3α-ursolic acid epimers present in fresh *L. orientalis* resins to their more stable 3β-epimers via their respective ketones? This may, in fact, be possible through a combination of two mechanisms an Oppenauer oxidation and a Meerwein-Ponndorf-Verley (MPV) reduction (as proposed by Dr W. Martin, **Figure 7.34**).

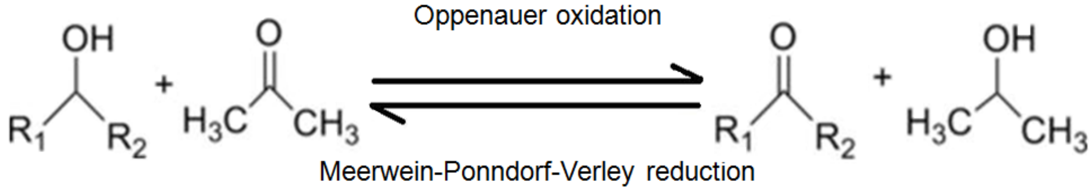


Figure 7.34. Equilibrium reaction catalysed by metal ions comprising an Oppenauer oxidation, the forward reaction, and Meerwein-Ponndorf-Verley (MPV) reduction, the reverse reaction.

These processes are catalysed by metal ions and are reversible. Thus, if such reactions were operative within the burial environment, equilibrium between the 3α- and 3β-epimers of oleanolic and ursolic acid and their respective ketones would be established. The position of equilibrium would then be dependent on the relative stabilities of the different epimers and temperature. The deposition of a balsamic resin initially containing the less stable 3α-epimers with their associated ketones would, therefore, given time, be expected to reach an equilibrium consisting of the ketones and both epimers. Indeed, as the more stable product seems to be favoured in such processes when they continue for extended periods, a greater abundance of the 3β-epimer might be predicted. Theoretically, this could account for the relative abundances observed in the Winchester and Poundbury samples with further oxidation in the latter producing the *O*-acetyl derivatives.

Thus, evidence for the inclusion of plant exudates was not recovered from the lead-lined coffin burial from St Martin's Close, Winnall (F57) but analysis of materials from the focal burial, G336, Eagle Hotel site, Andover Road, Winchester established the presence of biomarkers characteristic of a Pinaceae resin together with LMM phenolic compounds and a range of triterpenic isomers. Although clear identification of the source of the latter is still being sought, it seems probable that a balsamic exudate, possibly derived from *Liquidambar orientalis*, was also present.

7.6 Case Study 5: Boscombe Down, Wiltshire (Brettell *et al.* 2015a)

7.6.1 Background

In the pre-Roman period, the present county of Wiltshire appears to have been occupied by a number of ill-defined groups (Frere 1991: 39) with the north-east traditionally connected with the Atrebates, the north-west with the Dobunni and the south with the Durotriges. Incorporated within the province of *Britannia* during the western campaign of Vespasian (c. 45 AD), this region does not correspond to any known Roman administrative unit (Salway 1981: 43-44, 92-93). Archaeological evidence has provided few signs of military occupation or of substantial towns. The best attested semi-urban sites with planned layouts, public buildings and markets appear have been at Mildenhall, Wanborough and Stratford-sub-Castle/Old Sarum. A number of smaller settlements developed along the network of pre-existing and Roman roads, the latter constructed in the mid-late 1st century AD. Many villa estates (c. 50-80) have also been identified including a palatial complex at Castle Copse, sites with high quality mosaic floors and painted wall plaster (e.g. Box) and smaller farmsteads with luxury features (e.g. Littlecote) (Corney 2001; Draper 2006: 9-13; Griffiths 2001).

Thus, the general impression is of a rural landscape of scattered farms and small nucleated settlements surrounded by organised field systems (Frere 1991: 266). Significant continuity from the late Iron Age is indicated, with an agricultural-based economy supplemented by cattle, sheep and pig husbandry with local crafts and industries such as ceramic production, metalworking and quarrying (Draper 2006: 10-12; Fulford *et al.* 2006: 191-194). The stability this provided is reflected in an increased level of prosperity especially in the 3rd-4th centuries AD and can be traced in the gradual adoption of Roman material culture through the use of coins, imported ceramics and architectural features such as hypocausts (Draper 2006: 14-15; Fulford *et al.* 2006: 201-215). These artefacts seem to have had a limited distribution beyond the more urbanised areas (Fulford *et al.* 2006: 114-121).

After 350 AD, as elsewhere in Britain, a period of decline can be observed although accurate dating is problematic (Draper 2006: 27-29). Some villas seem to have been abandoned by c. 370 AD while others show signs of reduced or 'squatter' occupation. In contrast, a number of the smaller estates/farmsteads continued to flourish and substantial coin hoards have been discovered, perhaps reflecting the importance of the area as a supplier of grain to the army in the late 4th-early 5th century AD. By c. 450 AD, however, all evidence of a 'Romanised' lifeway disappears from the archaeological record (Draper 2006: 30-35; Griffiths 2001).

This intensively-exploited landscape required numerous burial grounds to serve its wide variety of settlements. As much of the evidence recovered from the region relates to individual finds or poorly-reported excavations, the record is somewhat piecemeal although this is changing with new research and publications (Foster 2001). What can be discerned is that, as in other areas of the south-west, crouched inhumation appears to have been the main mortuary practice in the late PRIA and continued into the early Roman period, in contrast to the adoption of cremation as the dominant rite elsewhere in the province (Philpott 1991: 8; Whimster 1981: 37-59). In fact, most burial grounds in Wiltshire contain a mixture of both cremation and inhumation burials with continuance of this pattern throughout the Roman period. With regards to those inhumed, during the 1st-early 2nd centuries AD uncoffined burial in a shallow grave seems to have been commonplace. This was superseded by extended, coffined inhumation in the late 2nd century AD. The latter tend to be variously orientated with little or no extant grave goods. Nonetheless, some evidence for more elaborate mortuary practices has been recorded in the form of small mausolea (e.g. Truckle Hill; Nettleton), stone-lined cist graves, large timber-lined vaults (e.g. Boscombe Down Sports Field), lead-lined coffins (e.g. Birchanger Farm, Westbury; Black Field, Mildenhall) and stone sarcophagi (e.g. Purton; Bradford-on-Avon; Iford Manor, Westwood; Parsonage Farm, Winsley) (Foster 2001).

Over the past fifteen years, extensive excavations on Boscombe Down (SU 163 400) have revealed the presence of at least five substantial burial grounds. When published, the results will add significantly to our understanding of life and death in Roman Wiltshire as over 300 burials have been recovered, including one individual in a substantial stone sarcophagus (Site Code 56246; Context 12821; Grave 12785). This find was located within a small square enclosure and seems to have been the focal burial around which the cemetery developed. Constructed of Chilmark limestone with an apsidal head-end, the sarcophagus had an ill-fitting capstone but was otherwise intact (**Figure 7.35a**). Once opened, it was found to contain the remains of an adult female (SK12787) and a c. seven year old child (SK12823). A small amount of fill had entered the sarcophagus through the void at the head-end but disturbance was minimal (**Figure 7.35b**).

No evidence of tumbling due to water-action or the activities of small mammals was observed, although coffin fly (*Megaselia scalaris*) puparia were present. Thus, although the unique micro-environment within the sarcophagus had resulted in extremely poor bone preservation the position of the bodies had been maintained and the decomposition of associated organic materials retarded. This had permitted the survival of the fur-lined, deer-skin slippers with cork insoles worn by the woman and the calf-skin shoes of the child. Dated to the early 3rd century AD, grave goods consisted of a small ceramic beaker (of Gaulish/Rhineland origin), a necklace of jet beads and a copper anklet (Wessex Archaeology 2007).

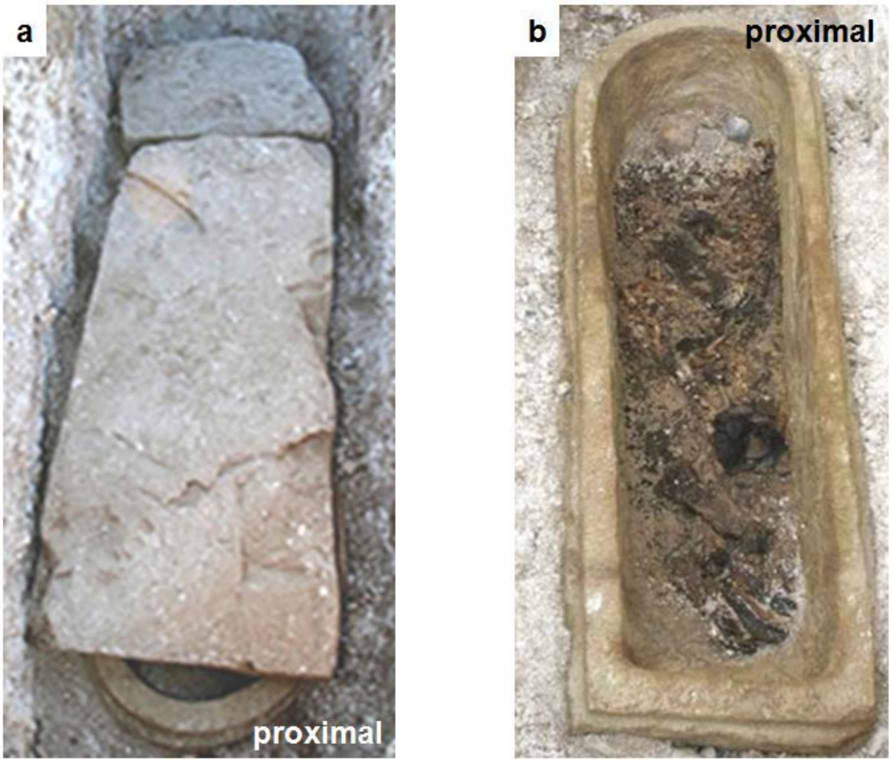


Figure 7.35. The Roman period sarcophagus, Boscombe Down, Wiltshire: a. with capstone in place, as found; b. the double burial and associated grave deposits (Images from Wessex Archaeology 2007).

7.6.2 Sample selection

Twenty-two samples were collected from the sarcophagus (Context 12821; Site Code 56246; Grave 12785) with the kind permission of Wessex Archaeology, Salisbury facilitated by Dr J. McKinley. These consisted of one sample of material associated with the cranium of the adult female, three samples of the lower density fraction of the water-floated grave deposits and eighteen samples of the higher density fraction (**Appendix 6.5**). All of these, with the exception of the material collected from the bag containing the cranium, had been stored under water since 2008. They were, therefore, left to stand in a desiccator over silica gel until dry (**Figure 7.36**). A vial of water was placed in each desiccator and also allowed to evaporate to dryness, then solvent rinsed for analysis to ascertain if any contamination had occurred as a result of this pre-treatment.

Figure 7.36. Desiccator containing samples from the Boscombe Down sarcophagus and a vial of water as a method control to assess contamination (Author).

7.6.3 Results

The samples were minimally soluble in the organic solvent. All (#22) contained similar lipid species in varying abundance with the lighter fraction, the flot samples (BD1-3) providing the broadest range of results. Images of each sample with corresponding TICs and tabulated results are provided in **Appendix 6.5; Disc 1, File 6.5.** Trace levels of phthalate plasticisers, modern contaminants probably derived from the storage containers, were present in the majority of the samples. No lipids were observed in the solvent washes of the water control vials. In the archaeological samples, the majority of the lipids identified were found to belong to a number of commonly observed classes. These comprised *n*-alkanes (C_{15-33}), *n*-alkanols (C_{17}-C_{30}), saturated ($C_{12:0}$-$C_{24:0}$) and unsaturated carboxylic acids ($C_{18:1;\ 18:2}$) together with steroidal compounds (cholesterol, cholesta-4,6-dien-3-ol, cholesta-3,5-dien-7-one, β-sitosterol) and the stanol, 5α-cholestanol.

Based on their odd-numbered molecular ions and fragmentation patterns, the early-eluting peaks were characterised as short-chain amines and amides with confirmation provided by the NIST mass spectral library. Other nitrogen-containing species may also be present and some HMM polycyclic heteroatoms (e.g. benzenesulfonamides). Nonetheless, many could not be securely identified, including the dominant compounds in these samples. These consisted of a triplet (1-3) eluting between 17.8 and 18.1 min alongside the bacterial marker, diploptene (4), and friedoolean-3-one (5), a defunctionalised triterpenoid with a migrated methyl group, found in the tissues of various plant families (Shan *et al.* 2013; **Figure 7.37; Table 7.17**).

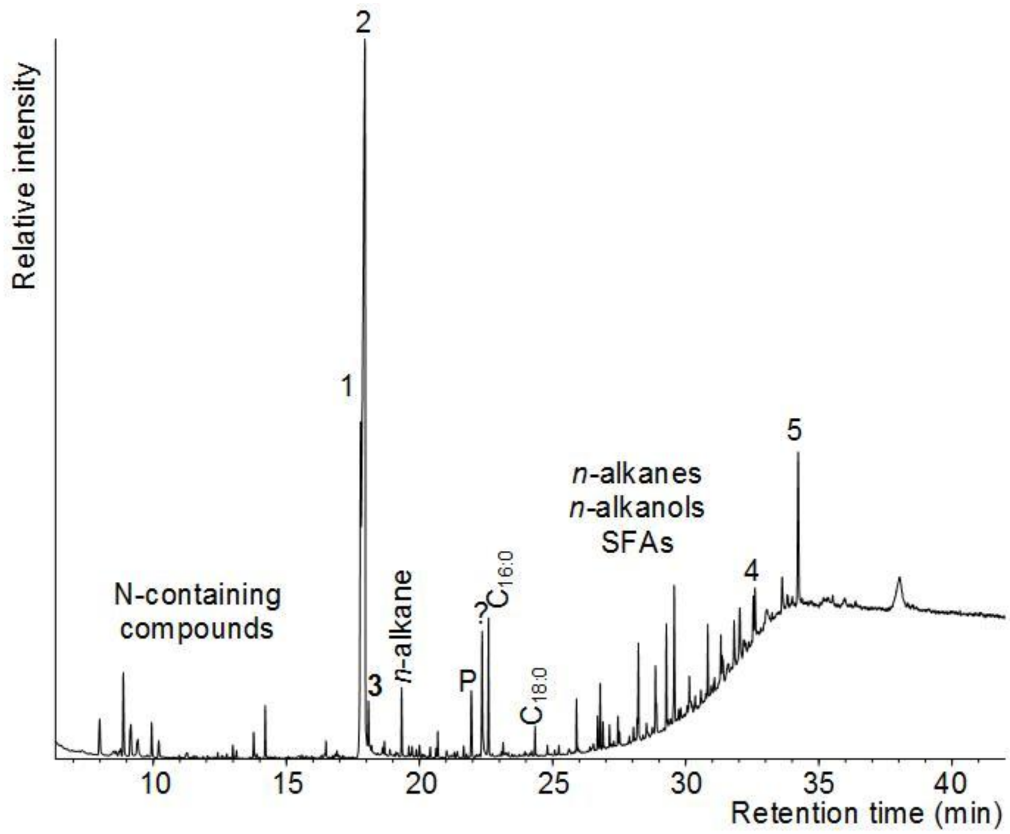


Figure 7.37. TIC chromatogram (BD2) representative of the grave deposits, sarcophagus burial, adult and child, Boscombe Down, Wiltshire, UK. Peak numbers relate to **Table 7.17**.

Table 7.17. Mass fragmentation patterns of most abundant compounds (TMS derivatives), grave deposits, stone sarcophagus, Boscombe Down, with identification where possible.

Peak	M^+ ion	BP	Key fragment ions	Name of compound
1	310	281	295, 268, 239, 209, 196, 139, 111, 73	?γ-lactone, phytenic acid
2	238	167	223, 209, 196, 153, 139, 111, 68	
3	238	153	223, 209, 196, 167, 139, 125, 69	
4	410	191	395, 383, 314, 257, 218, 121, 95, 69	diploptene
5	426	69	411, 302, 273, 218, 205, 191, 163, 123	friedoolean-3-one

Of the three unidentified moieties, Peaks 2 and 3 appear to be isomers due to their shared molecular mass and similar fragmentation patterns while Peak

1 may be a γ-lactone of phytenic acid produced by the microbial degradation of phytol (Simoneit *et al.* 1980). No terpenoids, characteristic of resinous exudates, were present.

7.6.4 Summary of findings

Similar results were obtained from all of the samples analysed with the range and patterning of the lipid species present atypical of most soil profiles. This corresponds with the physical evidence for minimal ingress from the surrounding burial environment. Evaluation of the source of the compounds identified indicates that some, such as the carboxylic acids, could derive from a variety of microbial, plant or animal sources (Evershed 2008a). Others, however, are more diagnostic with a microbial contribution denoted by the presence of LMM *n*-alkanes while those with longer carbon chains probably originated from the leaf waxes of higher plants (Kögel-Knabner 2002). The microbial decomposition of vegetative matter may also be the source of the long-chain *n*-alkanols with the sterol, *β*-sitosterol, and terpenoid, friedoolean-3-one, representative of an higher plant input (Jambu *et al.* 1993; Shan *et al.* 2013). In addition, some of the nitrogenous compounds could be natural alkaloids (e.g. pyrroles, (di)azoles and (di)azines) which are found in plants and fungi (Clayden *et al.* 2012: 1156-1161). These components may, therefore, derive from the decay of floral tributes and/or plant-derived textiles.

Many of the compounds present could also relate to the decomposition of the human remains and associated materials (e.g. leather). Chemical breakdown of the macromolecules found in mammalian tissues results in a wide range of degradation products (Gill-King 1997; Pfeiffer 1998). Some of the most common (e.g. SFAs) were observed in these samples with microbial decomposition of the human remains the most likely source of cholesterol and its derivatives. Likewise, in this instance, the presence of diploptene is probably a reflection of the bacterial degradation of bone (cf. Evershed *et al.* 1995) and is consistent with the poor preservation of the skeletal remains. The LMM nitrogenous compounds (although used in the detection of clandestine burials; Dekeirsschieter *et al.* 2009) are, however, an unexpected find in an archaeological context due to their volatility. It seems likely, therefore, that they result from post-excavation decay within the sealed containers of water of the comminuted materials (including bone) collected from the sarcophagus although this has not been experimentally ascertained.

Thus, the chemical traces remaining within materials from the base of the Boscombe Down sarcophagus appear to result from the degradation of intrinsic archaeological deposits. Accordingly, the absence of terpenoid biomarkers may indicate that resinous substances were not included within this Roman period double-burial. The excellent level of organic preservation, lack of disturbance and well-documented excavation policy lends some support to this contention. It should be noted, however, that natural variability (i.e. absence of HMM terpenoids from the exudates of some species) may prevent identification in the archaeological record (**8.5**) while post-excavation treatment (sieving and storage in water) could have removed other evidence (i.e. gum components) and introduced contaminants (e.g. plasticisers). Moreover, the effects of extended curation under water, which necessitated additional preparation procedures (i.e. desiccation), almost certainly impacted on the degradation pathways of the organic matter present in the samples and somewhat compromised the reliability of the data.

7.7 Case Study 6: Burial ground, Purton, Wiltshire

7.7.1 Background

As detailed in **7.6.1**, the current county of Wiltshire was largely rural in nature during the Roman period with few significant urban areas but many smaller settlements and villa estates (Corney 2001; Draper 2006: 9-13). These were supported by numerous burial grounds (Foster 2001). Thus, in 1987, contractors working at Northview Hospital, Purton (SU 0858 8736) uncovered evidence of Roman period remains (**Table 7.18**). Subsequent excavation by Thamesdown Archaeological Unit revealed a walled burial ground (Chandler 1994). This was probably linked to a villa complex due to its rural location although little archaeological evidence of Roman occupation in the Purton area has been recovered (Nurse 1992).

Table 7.18. Details of burials from Northview Hospital, Purton, Wiltshire, UK. Accession number: WILT MC 890036, Purton 806.

Grave	Type/date	Container	Osteology	Grave goods	Observations	Samples collected
1	Inhumation Early 4^{th} c. AD	Substantial grave cut Walled structure Stone sarcophagus Lead-liner Plaster body-casing	Gracile female 18-25 year old Additional facets on atlas; lumbar/sacrum fused; dental abnormalities	2 ceramic vessels; portion of shale bracelet; animal bones; high quality glass vessel; fine wool fabric with dyed border	Poor preservation: flaking of bone; brushite formation Dark-staining to post-cranial elements	PT1 residue/debris associated with femora PT2a + b debris associated with lead fragments PT3 debris associated with cranium
2	Cremation	Limestone ossuary Cylindrical lead urn Green glass vessel	Female c. 25-40 years old Additional facets on atlas	Part with a clear liquid (?water); organic residue floating on surface Cremated bird and animal bones; fragments of burnt ceramic + charcoal	Uneven level of cremation	PT4 residue associated with charred bone fragments PT5 cream-white residue PT6 clear liquid from vessel PT7 liquid from base of vessel PT8 liquid from top of vessel PT9 orange-grey residue PT10 cream-grey residue
3	Inhumation	Normative rite Disturbed by machinery No evidence of shroud or container	Robust adult male (50+) Activity-related markers; eburnation to medial clavicle; osteoarthritis of vertebrae – indicators of physical labour		Good preservation Incomplete	PT11 traces of soil adhering to left distal femur and proximal tibia
4	Inhumation	Grave cut only observed				NA
5	?Inhumation	Unrecovered bones observed by contractors				NA
6	Inhumation Late 3^{rd}- early 4^{th} c. AD	Stone sarcophagus	Gracile female Mature adult (40+) Osteoarthritic changes; periosteal new bone on ribs; additional facets on atlas; dental abnormalities	Black Burnished-Ware bowl with 'porridge-like' residue in base; ?4^{th} century AD coin; corroded iron fragments	Moderate preservation Near complete	PT12 dark residue adhering to hyoid PT13 dark residue adhering to cranium
7	Inhumation	Normative rite Disturbed by machinery No evidence of shroud or container	Robust adult male (30+) Activity-related markers, upper body – indicators of physical labour		Good preservation Incomplete	No residues or detritus remaining

Six inhumations were recorded which included two graves from which human remains were not recovered due to mechanical disturbance (Graves 4 and 5). Two others, also disturbed, contained the remains of individuals interred in a normative fashion, a mature (50^+) adult male (Grave 3) with skeletal markers indicative of heavy manual labour and severe osteoarthritis and a robust younger (30^+) male with prominent upper body muscle attachments (Grave 7). The two remaining inhumation burials contained the skeletons, possibly of females, who had been interred in stone sarcophagi. The first was a mature (40^+) adult, placed supine, extended and accompanied by a Black Burnished Ware bowl (late 3rd-early 4th century AD) and a heavily worn ?4th century AD coin placed near the mandible (Grave 6) (Chandler 1994). Six right ribs showed extensive periosteal new bone formation indicative of a lung infection although it could not ascertained if this contributed to her death (McKinley 1994).

The final example (Grave 1) was that of a young adult female (aged around 18-25 years; McKinley 1994) who had been interred in a sarcophagus with a lead-liner (Type 2, Toller 1977: 11; **Figure 7.38a**). This burial was located within a circular structure and had been placed in a four metre square grave which had been cut into the bedrock. The sarcophagus tapered towards the foot and had iron-stained sockets, two short iron bars attached externally to the right and left sides and three equidistant bars across its width (**Figure 7.38b**). Two ceramic vessels, a Black Burnished Ware pot (early 4th century AD) and an amphora together with a portion of a shale bracelet and animal bones (mostly ovicaprid) showing butchery marks were found within the grave cut. A high quality glass vessel (late 3rd-early 4th century AD), traces of plaster (not evident in the photograph), a considerable amount of fine wool tabby weave fabric with a coloured twined-cord border and hobnails, denoting footwear, were recovered from within the burial containers (Bond 1994; Chandler 1994; Walton and Wild 1994).

Figure 7.38. Grave 1, Northview Hospital, Purton, Wiltshire, UK: a. the lead-liner within the sarcophagus; b. the skeletal remains and iron cross bars (Costello 1987); c. the sticky yellow-brown residue, shaft of the left femur (Author).

It was reported that this gracile individual may have been embalmed (Nurse 1992). Examination of the post-cranial elements revealed a dark, shiny coating and poor state of preservation with delamination of the cortical bone and an abundance of brushite crystals in the trabecular bone (**Figure 7.38c**). The condition of the remains bore a striking visual resemblance to those from a late Roman period burial in Thessaloniki, Greece which had provided evidence of treatment with resinous substances (Papageorgopoulou *et al.* 2009; **Figure 3.2**).

In addition, an elaborate cremation burial of uncertain date was discovered (Grave 2; **Figure 7.39**). Osteological analysis showed that this represented the unevenly burnt remains of a mature adult (25-40 years old), possibly a female (McKinley 1994). She had been deposited within a blue-green glass vessel which had been placed in a cylindrical lidded lead urn decorated with a moulded bead and reel motif, scallop shells, a St Andrew's cross and two roughly inscribed crosses, one on the lid. The whole had then been set within an oolitic limestone ossuary and was accompanied by charred plant matter, animal (pig and ovicaprid) and bird bones (Bond 1994; Chandler 1994). All three of these possible females showed non-metric traits which may indicate a genetic relationship. These included additional facets on the superior surface of the atlas and, in the case of the two sarcophagi burials, additional cusps on the molars together with a rare molar root anomaly (McKinley 1994).

Figure 7.39. The Roman period lead urn containing a glass cremation vessel, Grave 2, Northview Hospital, Purton, Wiltshire, UK (Costello 1987).

Unusual deposits within the glass cremation vessel prompted chemical investigation as, on discovery, it was about half full of a clear liquid with a quantity of organic matter floating on the surface (Chandler 1994). In 1988 Raymond White found this residue to be an hydrolysed and saponified fat or oil with no indications for the presence of beeswax or resinous exudates. The ubiquitous nature of the compounds present meant that the precise source of this material could not be determined. Endogenous human marrow or body fat was considered unlikely unless substantially modified by bacterial action (White 1994). Nonetheless, as the overall level of cremation was poor, with evidence of incomplete combustion of the ligament/muscle tissue, this remained a possibility (McKinley 1994). A fat-based stopper that had fallen into the water was also suggested but no traces of this around the rim of the vessel were noted. Materials from the lead-lined sarcophagus were not assessed.

7.7.2 Sample selection

Thirteen samples, ten solid and three liquid (left to evaporate under a protective cover) were collected from four of these burials (Graves 1, 2, 3, and 6). Sample selection and mass was constrained by the materials available (**Appendix 6.6**). The skeletal remains had largely been cleaned for osteological analysis so no samples could be obtained from Grave 7 while the textiles from Grave 1 and grave deposits visible in the excavation photographs of Grave 6 could not be located (**Figure 7.40**). In the absence of suitable materials from elsewhere on site, an apparent soil sample was collected from the left distal femur and proximal tibia of the adult male interred in a normative manner in Grave 3 to act as an environmental control. Images of each sample with corresponding TICs and tabulated results are provided in **Appendix 6.6; Disc 1, File 6.6.**

Figure 7.40. Sarcophagus burial, Grave 6, Northview Hospital, Purton, Wiltshire, UK after removal of the lid during excavation showing extensive grave deposits (Costello 1987).

7.7.3 Results

7.7.3.1 Control sample, Grave 3 (the normative inhumation)

The control from Grave 3 was found to contain traces of *n*-alkanes and a range of phthalate plasticisers. The latter are modern contaminants and almost certainly derive from the plastic bag in which the remains had been stored. The *n*-alkanes could also have come from this source but this cannot be confirmed.

7.7.3.2 Sample details and results, Grave 6 (the sarcophagus burial)

The residue from the cranium had the appearance of clods of soil (rather than resembling the grave deposits visible in the excavation photographs) while that from the hyoid bone comprised flakes of dark organic matter. These samples from Grave 6 were dominated by phthalate plasticisers. *N*-alkanes, saturated (SFA) and monounsaturated (MUFA) carboxylic acids and *n*-alkanols (C_{12-32}, C_{26} maximum, even over odd) were also present in the material from the cranium (PT13). These are ubiquitous end products of the degradation of plant and/or animal tissues so cannot be ascribed a specific origin. The distribution of the *n*-alkanols, however, together with a vestige of campesterol supports some higher plant input while traces of an hopane (base peak *m/z* 191) may reflect a microbial contribution (Greenwood *et al.* 2006).

The sample from the hyoid (PT12), as with that from the 'Spitalfields Lady' (**7.4.3.1**), proved more illuminating as, due to its fragility, it had not been cleaned. The dark adhering matter contained an abundance of SFAs ($C_{6:0-20:0}$) dominated by $C_{16:0}$ with a considerable amount of $C_{18:0}$ and $C_{18:1}$ isomers. A number of α,ω-diacids (oxidised carboxylic acids) together with 10-hydroxyoctadecanoic acid ($10\text{-}OH_{18:0}$) and 10-oxooctadecanoic acid ($10\text{-}OXO_{18:0}$), hydration and oxidation products of cis-$C_{18:1\Delta 9}$ (oleic acid) respectively, were also observed. These moieties are indicative of the arrested decay of a lipid-rich substance, probably as a result of limited microbial action in waterlogged anaerobic conditions (Bull *et al.* 2009). Thus, the material from the hyoid represents a partially-degraded animal fat or plant oil.

7.7.3.3 Sample details and results, Grave 2 (the cremation burial)

With regards to Grave 2, the elaborate cremation burial, no lipids of archaeological relevance were detected in the dried residues from the three liquid samples (PT6-8). In contrast, a sub-sample of the layer of mixed bone fragments and associated amorphous matter floating on the liquid (PT4) displayed an abundance of the same combination of lipids observed in PT12 from Grave 6. This residue contained an homologous series of SFAs ($C_{6:0\text{-}20:0}$) dominated by $C_{16:0}$ with $C_{18:1}$ isomers and their microbially-mediated oxidation products (10-$OH_{18:0;}$ 10-$OXO_{18:0}$) (**Figure 7.41**).

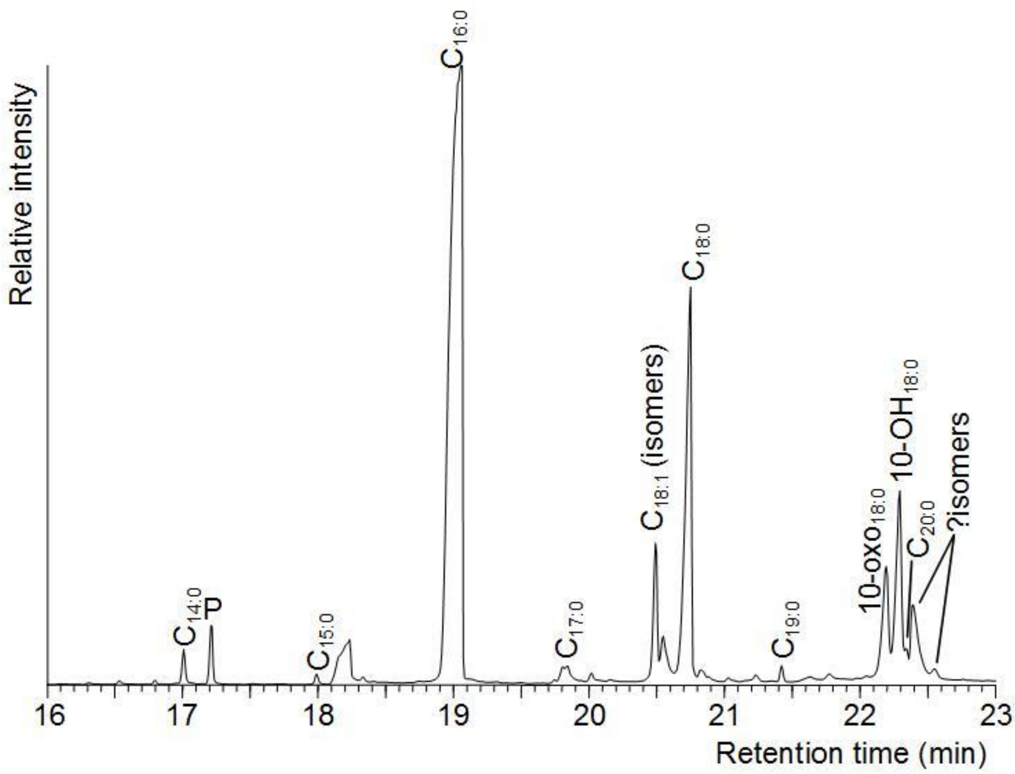


Figure 7.41. Partial TIC (16-23 min) residue associated with charred bone (PT4), cremation burial, Grave 2, Purton, Wiltshire, UK.

The remainder of the samples from the cremation burial (PT5, PT9, PT10) also contained an abundance of carboxylic acids ($C_{6:0\text{-}18:0,}$ $C_{16:0}$ max, $C_{18:1}$) although derivatisation was hindered by some intrinsic factor. This issue persisted, creating broad fronting peaks, despite the samples having been thoroughly dried prior to analysis with replicates run after re-derivatisation. One possibility is that interference by metal ions leached from the cremated bone and/or glass container had resulted in partial saponification of the lipids present thereby preventing TMS groups from attaching to any hydroxyl (-OH) and carboxyl (-COOH) functionalities. Nonetheless, it is clear that this material denotes the deposition of an animal fat or plant oil within the cremation vessel, as reported by White (1994).

In addition, these residue samples (#4) from Grave 2 contained traces of β-sitosterol, stigmasta-3,5,-dien-7-one and the defunctionalised triterpenoid, friedoolean-3-one (10), indicative a higher plant input together with a series of wax esters (C_{32-36}) denoting the presence of an epicuticular leaf wax in PT10. Triterpenes with oleanane and ursane-skeletons and a double bond at C-12 were also identified based on their characteristic fragmentation patterns (significant ions at *m/z* 218, 203 and 189) (**Figure 7.42; Table 7.19**).

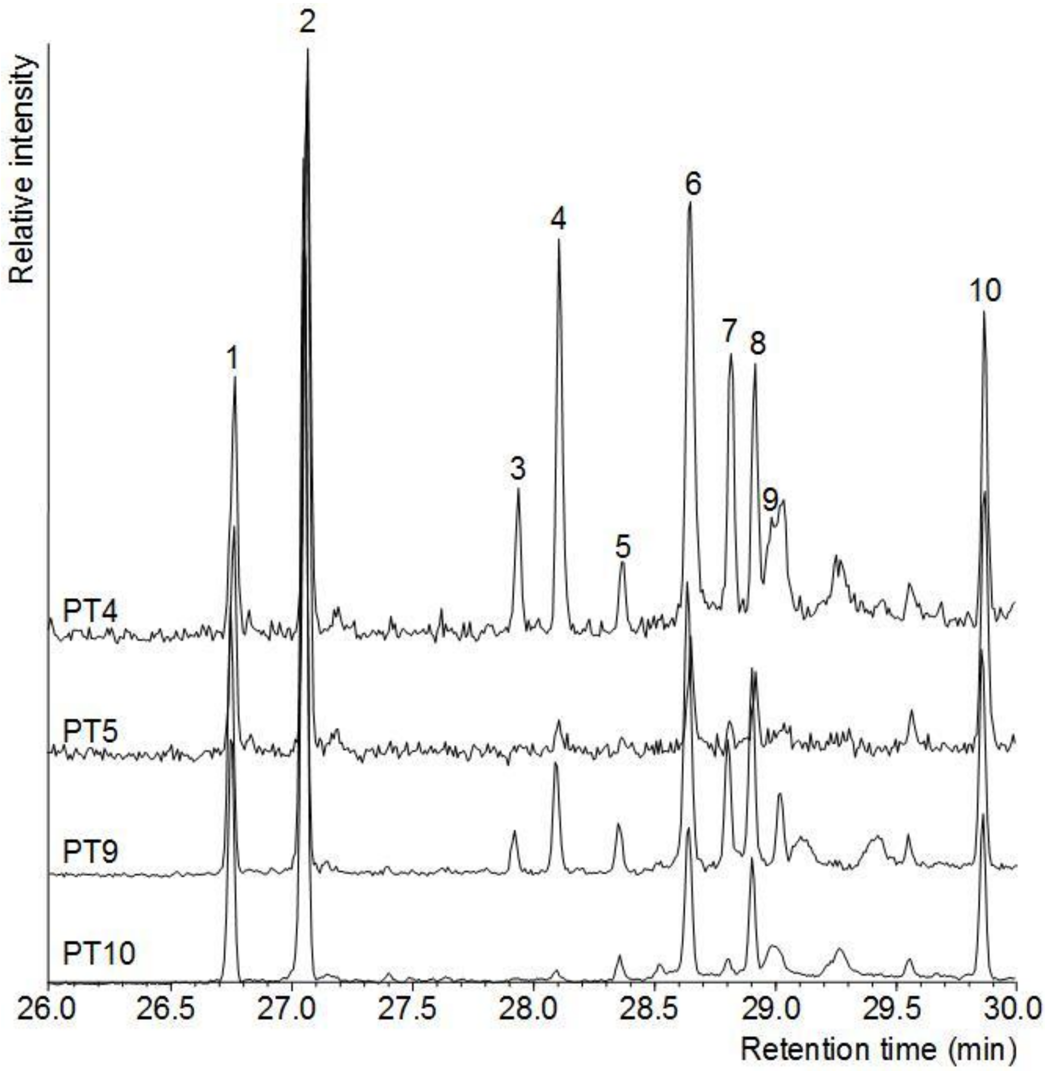


Figure 7.42. XIC (*m/z* 218) amorphous residues, cremation burial, Grave 2, Purton, Wiltshire, UK. Peak identifiers relate to **Table 7.19**.

Table 7.19. Identification of terpenic compounds (TMS derivatives), residues, cremation burial, Grave 2, Purton, Wiltshire, UK based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
1	394	218	369, 203, 189, 175	24-norolean-3,12-diene
2	394	218	379, 203<189, 175, 119, 107	24-norursa-3,12-diene
3	498	218	408, 292, 203>190/189, 175, 133	3α-hydroxy-olean-12-en-3-ol (3-*epi*-β-amyrin)
4	498	218	483, 239, 203=189<190, 133	3α-hydroxy-urs-12-en-3-ol (3-*epi*-α-amyrin)
5	?	218	203>189	oleanane derivative
6	408	218	393, 257, 232, 203>189, 175, 119	24-noroleana-3,12-dien-11-one
7	410	163	395, 218, 203, 191, 133, 119, 105	28-norolean-17-en-3-one
8	424	218	409, 231, 203<189, 175, 163	urs-12-en-3-one (α-amyrenone)
9	498	218	483, 408/9, 279, 203<189, 135	3β-hydroxyurs-12-en-3-ol (α-amyrin)
10	426	69	411, 341, 302, 273, 246, 205, 163	friedoolean-3-one

The low abundance and highly degraded nature of these compounds makes secure taxonomic assignment problematic. Nonetheless, this combination of olean-12-enes and urs-12-enes with the greater abundance of the latter, particularly in terms of the pentacyclic alcohols which displayed a ratio of approximately 2:1 (3-*epi*-α-amyrin (4):3-*epi*-β-amyrin (3)), suggests a member of the Burseraceae (Stacey *et al.* 2006). This relationship has been observed in *Boswellia* spp. exudates and it appears that this may be the source in this instance. Indeed, the 24-nortriterpenes present have previously reported in heavily aged and/or thermally degraded frankincense and are considered to be diagnostic derivatives of the boswellic acids (Baeten *et al.* 2014; ten Haven *et al.* 1992; van Bergen *et al.* 1997).

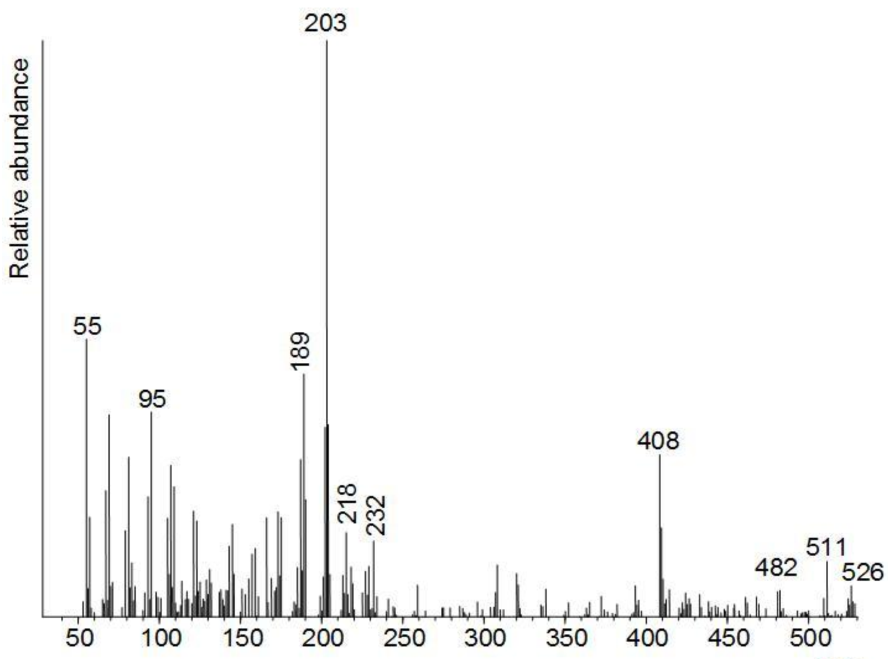


Figure 7.43. Mass spectrum (30.0 min) assigned as oleanonic acid, amorphous residue (PT4), cremation burial, Grave 2, Purton, Wiltshire, UK.

The absence of these resins acids in the Purton samples prevents clear confirmation while the presence of 28-norolean-17-en-3-one (7) and, in PT4, what appears to be oleanonic acid (**Figure 7.43**) adds doubt. These compounds have not been reported in *Boswellia* spp. gum-resins. A second contributor, such as a *Pistacia* spp. resin or *L. orientalis* exudate, is, therefore, a possibility since oleanonic acid is of relatively restricted occurrence while 28-norolean-17-enes are stable end products formed by decarboxylation of oleanane-skeleton acids with subsequent migration of the double bond (Pastorova *et al.* 1998; ten Haven *et al.* 1992).

7.7.3.4 Grave 1, lead-lined sarcophagus burial

The samples from Grave 1 (PT1-3) were also found to contain a series of SFAs ($C_{6:0\text{-}18:0}$), dominated by $C_{16:0}$, branched chain SFAs ($C_{15:0\text{-}17:0}$) and MUFAs ($C_{16:1;}$ $C_{18:1}$ isomers) together with cholesterol and a number of derivatives (**Figure 7.44**).

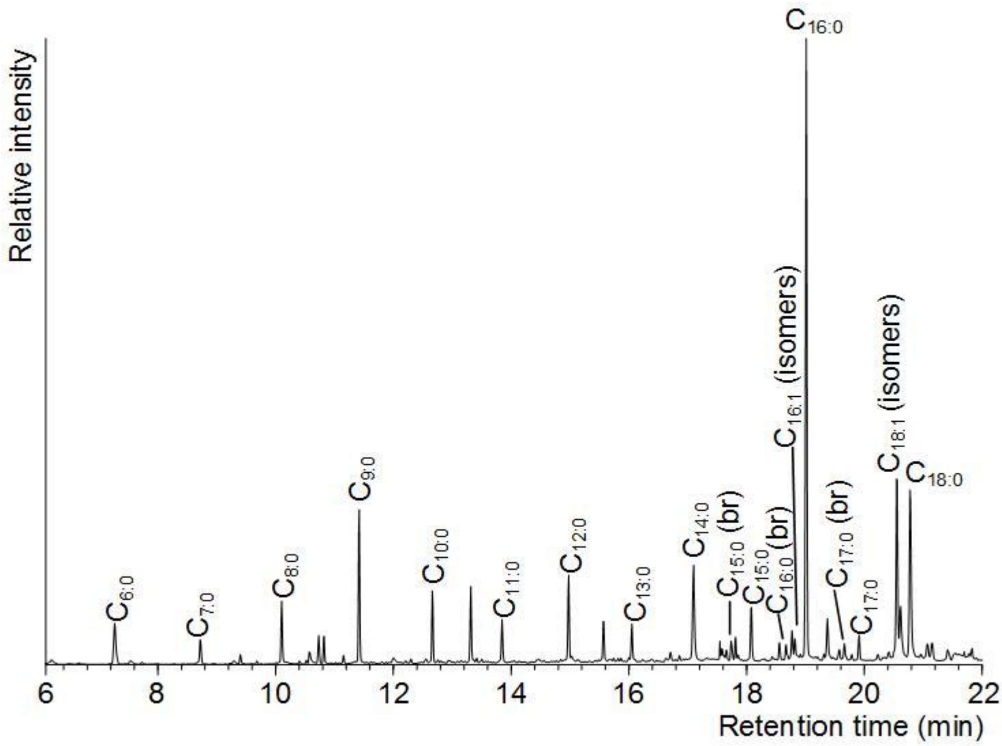


Figure 7.44. XIC (*m/z* 117) carboxylic acids, residue (PT1), sarcophagus burial, Grave 1, Purton, Wiltshire, UK.

These suggest a mammalian input (degraded body tissue or introduced animal fat) although no di- or hydroxyl acids were observed. The bimodal distribution of the SFAs (lower maximum at $C_{9:0}$), relative abundance of the

LMM odd carbon-chain moieties and presence of branched chain SFAs may indicate microbial action but could equally denote a ruminant origin (c.f. Evershed *et al.* 1997b). The *n*-alkanols (C_{26} maximum), traces of phytosterols (campesterol and β-sitosterol) and HMM esters ($C_{40\text{-}46}$) derived from the epicuticular leaf waxes of higher plants in PT1 support a contribution from degraded plant matter.

Diterpenic (PT1; PT3) and triterpenic compounds (all samples) were also observed. The diterpenoids were found to have abietane and pimarane skeletons based on their characteristic fragmentation patterns and indicate the inclusion in this burial of a resin from a member of the Pinaceae family (**Figure 7.45; Table 7.20**).

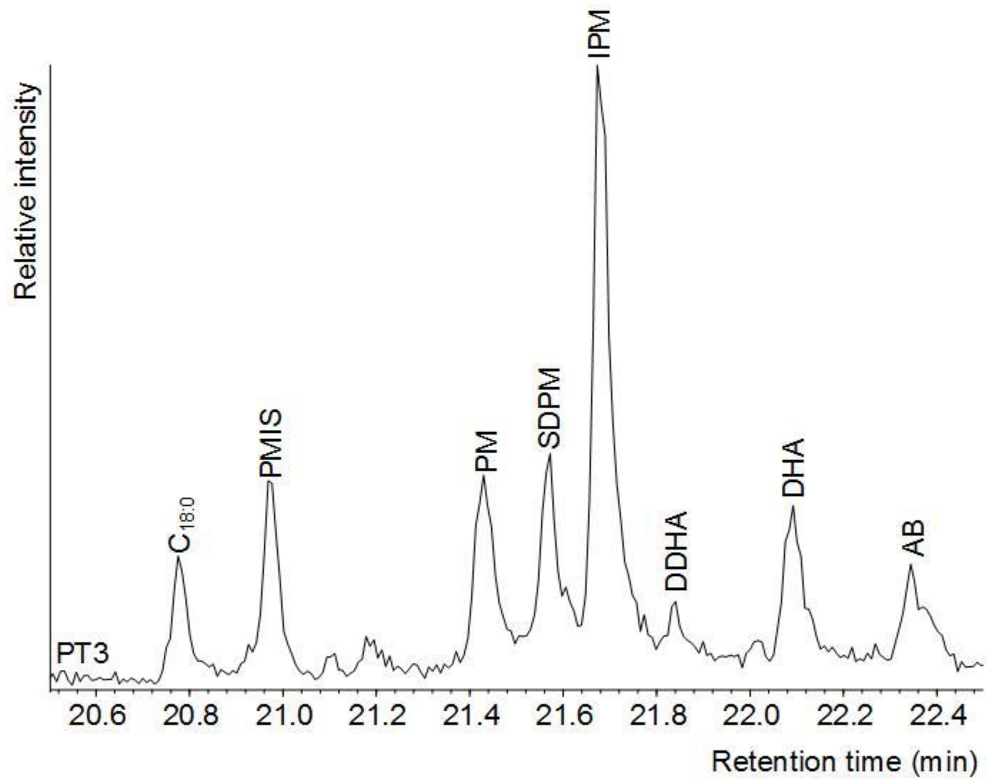


Figure 7.45. Partial XIC (m/z 241) diterpenic compounds, residues (PT3), sarcophagus burial, Grave 1, Purton, Wiltshire, UK. Peak identifiers relate to **Table 7.20**.

Although only traces remained, this exudate was relatively well preserved as the resin acids, pimaric (PM), sandaracopimaric (SDPM), isopimaric (IPM) and abietic (AB) acid, were detected. Some degree of natural aging was also evident due to the presence of functionalised derivatives such as DHA, DDHA and a trace of 7-oxodehydroabietic acid (23.7 min). Defunctionalised markers of extensive thermal alteration (e.g. retene) were not observed. These observations were supported by comparison with modern Pinaceae products including *Pinus pinaster* and *Pinus sylvestris* resins and a *Pinus sylvestris* tar (**Appendix 3.1**).

Table 7.20. Identification of diterpenic compounds (TMS derivatives), residues (PT3), Grave 1, Purton, Wiltshire, UK based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
PMIS	374	241	359, 332, 257, 256, 173, 91, 73	unidentified pimarate isomers
PM	374	73	359, 299, 257, 207, 191, 133, 121	pimaric acid
SDPM	374	121	359, 257, 241, 157, 143, 105, 73	sandaracopimaric acid
IPM	374	241	359, 257, 256, 143, 105, 73	isopimaric acid
DDHA	370	237	355, 252, 209, 195, 143, 103, 73	didehydroabietic acid
DHA	372	239	357, 255, 240, 185, 143, 129, 73	dehydroabietic acid
AB	374	256	257, 241, 213, 185, 105, 73	abietic acid

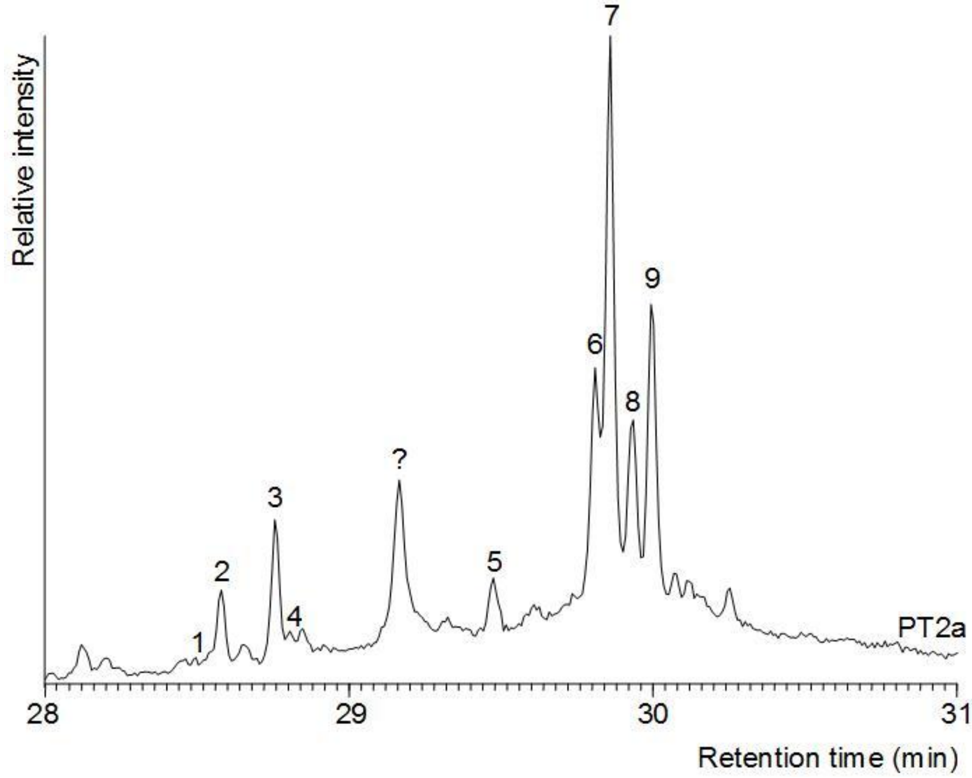


Figure 7.46. Partial XIC (*m/z* 203) triterpenic compounds, residues (PT2a), Grave 1, Purton, Wiltshire, UK. Peak identifiers relate to **Table 7.21**.

Table 7.21. Identification of triterpenic compounds (TMS derivatives), residues (PT2a), sarcophagus burial, Grave 1, Purton, Wiltshire based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
1	410	204	395, 381, 313, 245, 215, 189, 175, 133	28-norolean-12-en-3-one
2	424	218	409, 218, *203 much>189,* 122, 95, 55	β-amyrenone
3	410	163	393, 279, 257, 218, 203, 133, 119	28-norolean-17-en-3-one
4	408	408	393, 379, 231, 218, 203, 189, 173, 129	28-norolean-12,17-dien-3-one
5	422	422	407, 216, 203/2, 189, 175, 161, 119, 105	?oleanadien-3-one
6	526	189	511, 409, 391, 320, 307, 219, 203, 133	moronic acid
7	526	203	511, 408, 393, 320, 307, 219, 189, 133	oleanonic acid
8	438	203	409, 320, 232, 189, 175, 133, 119, 105	oleanonic aldehyde
9	600	203	585, 510, 482, 392/3, 320, 279, 189, 133	oleanolic acid

The triterpenic compounds, present in all of the samples collected from Grave 1, were found to have oleanane, tirucallane and dammarane skeletons based on their mass spectral fragmentation patterns. The key triterpenoids were identified as moronic (6) and oleanonic (7) acid (**Figure 7.46; Table 7.21**) with traces of *iso*masticadienonic (30.7 min) and masticadienonic acid (31.1 min) observed in PT2a (**Figure 7.47**).

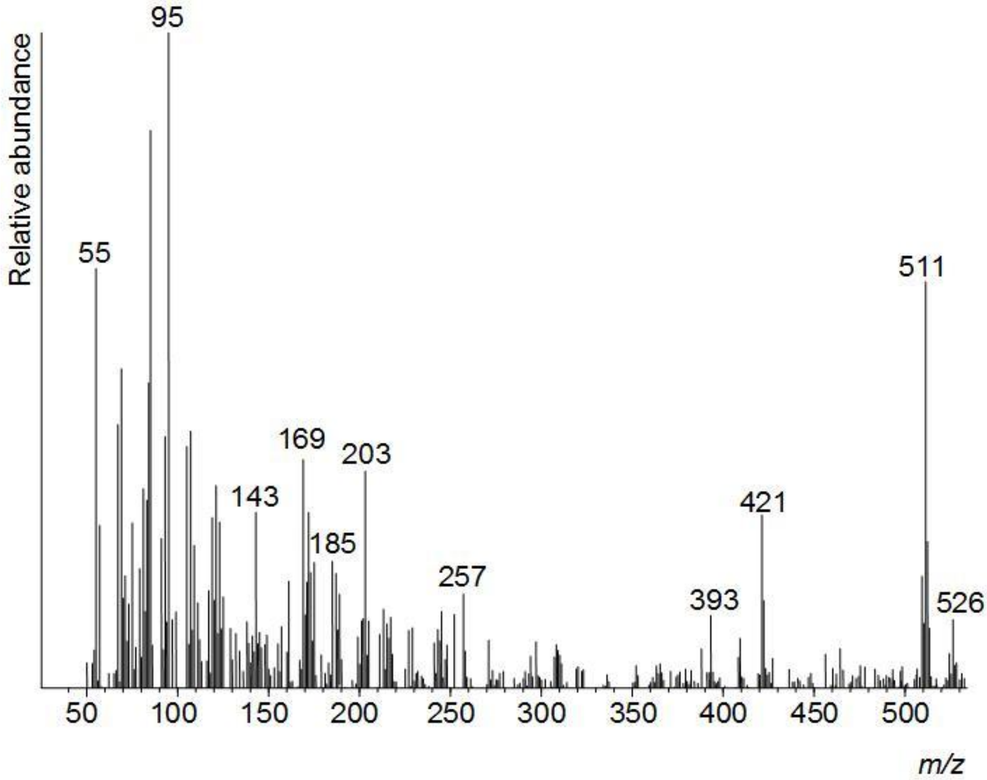


Figure 7.47. Mass spectrum (31.1 min) ascribed as masticadienonic acid (PT2a), Grave 1, Purton, Wiltshire, UK.

These compounds are biomarkers for resins from the genus *Pistacia* (**5.3.3**). The relatively low abundance of 28-norolean-17-en-3-one (3) and survival of 28-nor-17,12-oleandien-3-one (4) and 28-nor-12-olean-3-one (1) suggests that this resin was relatively well preserved. It had, therefore, probably not been heated (or not to a significant degree) prior to deposition although evidence of more extensive environmental degradation, in the form of ocotillones (base peak *m/z* 143), was observed in sample PT2a. Comparison with modern *Pistacia* spp. exudates **Appendix 3.2)** confirmed this identification and related observations.

7.7.4 Interpretation of findings and summary

The samples collected from the Roman burials at Purton, Wiltshire permitted direct comparison between residues associated with a normative inhumation (Grave 3) and those from two sarcophagus burials (Graves 1 and 6) and a multi-container cremation burial (Grave 2) from the same site. The results indicate that the array of lipid species present in the residues from the more substantial containers can be considered of archaeological relevance as they were absent from the sample selected as a control from Grave 3. These lipids comprised suites of markers characteristic of a degraded fat or oil in the three more elaborate burials. In addition, terpenic compounds signifying the inclusion of resinous exudates within the lead-lined sarcophagus of a young adult female (Grave 1) and incorporated with the cremated remains in a glass vessel placed in a lead ossuary (Grave 2) were identified.

7.7.4.1 Fatty matter: mammalian or plant origin

The survival of an extensive range of carboxylic acids, their derivatives and steroidal compounds within the samples from the two sarcophagus burials, Grave 1 and Grave 6, is almost certainly related to the protection afforded by these containers. These moieties reflect the arrested decay of lipid-rich organic matter although their precise origin is more difficult to identify as the degradation pathways of both mammalian fats and plant oils result in similar end-products whose relative abundances are highly variable and unreliable indicators as to source. Thus, the components observed could derive from any, or a combination of, the following:

1. degradation of the body tissues of the deceased;
2. treatment of the body through the application of oils/unguents;
3. textile wrappings impregnated or infused with an oil/fat-based balm;
4. libations poured into the coffin prior to the sealing of the grave.

In Grave 1, where extra protection was afforded by the lead-liner, the presence of significant levels of cholesterol and its derivatives provides support for a mammalian origin. The carboxylic acid profile could denote either human body fat or an applied ruminant fat modified by microbial action.

In Grave 6, the organic matter adhering to the hyoid appears to have been transformed into adipocere. This waxy substance derives from the incomplete decomposition of adipose tissue providing hydrolysis and hydrogenation outstrip liquefaction (den Dooren de Jong 1961). The resultant mixture may then persist for centuries as a semi-solid, stable mass due to the inherent acidic and hydrophobic nature of its components (Fiedler *et al.* 2009; Pfeiffer *et al.* 1998).

Diagnostic evidence for the presence of adipocere is provided by the depletion of $C_{18:1}$ and other unsaturated carboxylic acids and a substantial increase in $C_{16:0}$ (Evershed 1991; Forbes *et al.* 2004). Hydroxy acids and their corresponding oxo acids may then form providing later stage markers as the adipocere becomes more brittle and crumbly (Takatori 1996). This combination of compounds is present in the residue adhering to the hyoid of the individual interred in Grave 6. Definitive identification of source remains problematic, however, as transformation mechanisms still require elucidation with chemical composition seeming to vary in relation to the bacteria present, nature of the depositional environment and anthropogenic treatment of the body (Forbes *et al.* 2005a-c). Moreover, the relationship between the hydrogenation of $C_{18:1}$, formation of 10-hydroxyoctadecanoic acid, and predominance of $C_{16:0}$ has neither been satisfactorily explained nor thoroughly quantified whilst identification to species has proved elusive due to similarities in decomposition pathways and the ubiquity of carboxylic acids in nature (Forbes *et al.* 2005d). Nonetheless, given the context of this residue, human adipose seems the most likely source in this instance.

The cremation burial, Grave 2, likewise provided evidence for a transformed fat or oil in the form of a considerable abundance of an off-white substance found floating on the surface of the liquid associated with the cremated remains. The multiple containers used in this elaborate rite effectively provided a sealed context (with the possible exception of water ingress) and so the organic components can be considered of archaeological significance. Theoretically, the four hypotheses listed above could again apply here as the remains showed indications of variable heating (McKinley 1994). It is interesting, therefore, to note that no evidence of cholesterol or related compounds was observed although phytosterols and epicuticular leaf wax esters (C_{32-36}) were present. This makes a source exogenous to the body, such as a plant oil, a strong contender. Even given the relatively low temperature of the burn (McKinley 1994), it seems unlikely that the fatty components of an anointing oil or unguent applied to the body or the textiles could have survived the process of cremation in such abundance. Indeed, they are more likely to have acted as an accelerant which suggests that the substance was added to the cremated remains after the latter had been gathered for burial. As a range of offerings and libations appear to have been deemed acceptable in this context (**2.3.2**) both the watery liquid and the organic matter could derive from deliberate deposits.

One interesting possibility is milk (with the plant-derived markers from another source) which effectively comprises a natural emulsion of saturated and unsaturated carboxylic acids in ~87% water (Månsson 2008). Over time these fractions separate into 'curds and whey'. Waterlogged, low oxygen conditions within the glass vessel could then have led to the transformation of the lipid components (curds) into an adipocere-like substance and have arrested further degradation. The range and relative abundances of the carboxylic acids present certainly fits those reported in milk with a predominance of $C_{16:0}$ and relatively high levels of $C_{18:0}$, $C_{14:0}$ and $C_{18:1}$ (c.f. Månsson 2008). Nonetheless, this combination could also have resulted from the breakdown of adipose tissues or even certain plant oils (e.g. olive oil) while evidence of cholesterol might be expected if this was, indeed, milk.

7.7.4.2 Terpenic evidence: plant exudates

The triterpenes identified in the samples from the cremation, Grave 2, support the incorporation of plant-derived matter within the glass vessel. Many of the compounds identified (e.g. the pentacyclic alcohols and their derivatives) are found in the tissues of angiosperms and are of widespread occurrence. The predominance of ursane-skeleton moieties and presence of 24-norterpenes indicates a member of the Burseraceae, probably frankincense (*Boswellia* spp.), alongside traces of a second exudate containing oleanonic acid (e.g. *Pistacia* spp. or *L. orientalis*). The low abundance and highly degraded nature of these resinous substances together with the absence of diagnostic resin acids and increase in defunctionalised and aromatised components is indicative of extensive oxidation possibly due to pyrolysis. These aromatic substances may have been cremated with the body or burnt as incense and their ‘ashes’ added to the urn. Nonetheless, an abundance of fatty matter, perhaps a plant oil, suggests that post-burn deposition of a scented oil/unguent or a combination of offerings (e.g. resin(s), plant oil, milk and/or water) is more likely.

The results from Grave 1, Purton are less complex to interpret. The diterpenic compounds present demonstrate the inclusion of a Pinaceae resin. Although in low abundance, the survival of the resin acids accompanied by a range of derivatives indicates natural aging. This is supported by the fact that the highly persistent end products of thermal alteration (e.g. methyl dehydroabietate and retene) were not observed. In addition, triterpenoid biomarkers for resins from the genus *Pistacia* were identified. The range and relative abundances of these compounds again implies relatively good preservation. Proposed markers of heating such as 28-norolean-17-en-3-one and other neutral compounds were in low abundance or absent. Thus, a mixture of unheated *Pistacia* spp. and Pinaceae resins seems to have been used in the mortuary rites accorded this young adult female.

7.7.4.3 Summary of findings

To summarise, extant samples from three inhumations (one providing a control) and an urned cremation from the late Roman rural burial ground at Northview Hospital, Purton, Wiltshire, UK were analysed. Both diterpenoid and triterpenoid biomarkers were identified in Grave 1 showing that this young adult female had been interred in a lead-lined stone sarcophagus accompanied by Pinaceae and *Pistacia* spp. resins, textiles and high quality grave goods. In addition, traces of triterpenic compounds, possibly indicative of *Boswellia* spp. (frankincense) and *Pistacia* spp. or *L. orientalis* exudates associated with a fatty/oily residue were present within Grave 2, the elaborate multi-container cremation burial.

7.8 Case Study 7: Burial grounds around Dorchester (Brettell *et al.* 2015a)

7.8.1 General background

Dorchester (Roman *Durnovaria*), Dorset, UK (SY 690 906) was established shortly after the Roman conquest with a planned civilian settlement laid out c. AD 60-65. Situated at an important road junction beside the River Frome, this site became the *civitas* capital of the *Durotriges* and, during the 2nd-4th centuries AD, saw the construction of an aqueduct, public baths, defensive circuit, amphitheatre (at Maumbury Rings) and a number of substantial town houses (Trevarthen 2008: 6-9, 14-30; Woodward *et al.* 1993: 359-367). The network of approach roads around Dorchester seems to have been lined with small settlements and farmsteads (Davies *et al.* 2002: 197) which operated in a profitable symbiosis with the urban market (Woodward *et al.* 1993: 367-375). After AD 350, Dorchester, like many other Roman towns, underwent of period of change and effectively ceased to function during the early 5th century AD (Esmonde Cleary 1989: 64-85; Sparey Green 1987: 65-69; Trevarthen 2008: 9-10, 31-43).

Following Roman law, the burial grounds of Dorchester were located outside the town walls (Startin 1982; Woodward *et al.* 1993: 367). The most extensive was situated to the west at Poundbury and probably served both urban and rural populations (Farwell 1993: 14-82; Sparey Green 1982). Modern construction work has also revealed evidence of Roman period graves at a number of other sites around the town including that at Crown Buildings (Sparey Green *et al.* 1982). Likewise, to the south-east, at Alington Avenue, the burial ground of a small rural community has been identified (Davies *et al.* 2002). Excavations in this *Durotrigian* tribal area have shown that crouched inhumation was the principal rite in the late pre-Roman Iron Age (PRIA) and remained dominant throughout the early Roman period in contrast to the adoption of cremation burial elsewhere. This local tradition was, however, superseded by extended inhumation during the 2nd-3rd centuries AD (Philpott 1991: 54-55). Subsequently, individuals were generally interred supine in wooden coffins with later developments characterised by their arrangement in orderly rows, WE aligned, with minimal grave goods (Farwell 1993: 6-82; Woodward 1993: 219-222). Late 4th century AD cist burials provide the final evidence prior to the establishment of a 5th-6th century AD settlement (Sparey Green 1982).

7.8.2 Sample selection

Thirty-seven samples were collected from twenty-two late Roman inhumation burials from around Dorchester, Dorset, UK (**Table 7.22**). Curated by Dorset County Museum (DCM; accession codes: 1994.5 Poundbury; 1991.89 Alington Avenue; 1998.38 Crown Buildings), residues adhering to skeletal elements, the inner surfaces of the plaster packing and mineral-replaced textiles as well as grave deposits were selected for analysis. One sample from the outer surface of the plaster packing of Burial 8, Poundbury, was collected to provide a control as, unfortunately, no soil or other environmental samples were available. Access to the skeletal remains from Poundbury, held at the Natural History Museum, London was also requested but denied as the author had not produced a publication on the analysis of residues adhering to skeletal materials at the time and the Arrington paper (Brettell *et al.* 2014) was deemed insufficient by the curator. For samples images and details of each set of results see **Appendix 6.7; Disc 1, File 6.7.**

7.8.3 Results

7.8.3.1 General information

A number of samples (#8) were not solvent extracted once they had been accurately weighed in the laboratory as they were below the required mass (**6.2**). Phthalate plasticisers were present in all of the samples analysed. These modern contaminants were particularly abundant in the materials obtained from the plaster body-casings from Poundbury. Presumably derived from the plastic bags, containers and sheeting used to store the human remains and associated materials, they appear to have been preferentially adsorbed within the matrix of the plaster. In addition, three samples did not contain peaks representative of other lipid moieties and so, if any organic compounds were present, they fell below the limits of detection. The remainder are discussed by site, below.

Table 7.22. Inhumations from Roman period burial grounds, Dorchester, Dorset, UK containing plaster and/or textiles assessed/sampled as part of this study *NB: Age is as given in the publications referenced; standard body position was supine, head to the west, legs extended, arms at sides; plaster is used as a general term where the white material present has not been analysed; *results of Raman spectroscopic analysis undertaken by Eline Schotsmans (2013).*

Grave	Age	Sex	Body position	Outer	Inner	Fill	Location	Textiles/grave goods	Samples taken
Poundbury; Accession Number 1994.5; SY 685 911; Farwell and Molleson 1993; Sparey Green 1982									
Grave 8	---	---	Standard (Reburied in situ)	Ham Hill	---	body-casing *gypsum	Main late Roman R2 mausoleum	Textile impressions Fragments of bone comb	*PD8 G1outer surface, control* PD8 G2, inner surface of plaster G3-5, residues adhering to plaster
Grave 49	9	?	standard	wood	---	some plaster *calcite (lime)	Main late Roman Site A	Plain weave cloth Textile impressions	PD49, dark residues adhering to plaster with textile impressions
Grave 99	50	M	standard	Ham Hill	---	body-casing plaster	Main late Roman R7 mausoleum	Textile impressions Gold fibres by foot	PD99a, associated debris PD99b yellow-orange fragments
Grave 127	15	F	standard	wood	lead	traces *lead substituted carbonate	Main late Roman Site E	Textile impressions	PD127a/b, dark residues adhering to plaster/mineral-replaced textiles
Grave 513	30	M	standard left hand on pelvis	wood	lead	body-casing plaster	Main late Roman R10 mausoleum	Textile impressions Basket weave cloth	PD513, dark residues adhering to plaster with textile impressions
Grave 517	35	F	standard	Ham Hill	---	body-casing *gypsum	Main late Roman R10 mausoleum	Bone comb Cu alloy ring	PD517, dark residues adhering to plaster with textile impressions
Grave 529	40	F	Standard hair preserved	wood	lead	body-casing plaster	Main late Roman R9 mausoleum	Textile impressions	PD529, orange residues adhering to plaster/mineral-replaced textiles
Grave 530	30	M	standard hair preserved	wood	lead	body-casing *gypsum	Main late Roman R9 mausoleum	Textile impressions Tabby weave cloth Wool headband, ?dyed	PD530a-b, d, dark residues on plaster near textile impressions PD530c orange residues on plaster
Grave 599	5	?	standard	wood	---	some plaster	Main late Roman Site B	Textile impressions	PD599 dark residues adhering to plaster with textile impressions
Grave 658	19	?	standard hands on abdomen	wood	---	some plaster	Main late Roman Site E	Textile impressions; 4th C coin; ring-headed pin	PD658 dark residues adhering to plaster with textile impressions
Grave 775	25	M	standard	wood	---	plaster in fill *calcite (lime)	Main late Roman Site E	Textile impressions	PD775 dark residues adhering to plaster with textile impressions
Grave 817	≥25	F	standard hair preserved	wood	lead	body-casing *lead substituted carbonate	Main late Roman Site E	Textile impressions	PD817 dark residues adhering to plaster with textile impressions
Grave 858	45	F	standard	wood	lead	body-casing *lead substituted carbonate	Main late Roman	Textile impressions	PD858 dark residues adhering to plaster with textile impressions
Grave 862	35	F	standard right hand on abdomen	wood	lead	body-casing plaster	Main late Roman Site E	Textile impressions	PD862 dark residues adhering to plaster with textile impressions
Grave 867	5	---	standard	wood	lead	body-casing *calcite (lime)	Main late Roman Site E	Textile impressions	PD867 dark residues adhering to plaster with textile impressions
Grave 868	4	---	standard	wood	lead	body-casing plaster	Main late Roman Site E	Textile impressions	PD868 dark residues adhering to plaster with textile impressions

Grave 892	40	M	standard right arm 90˚ left on abdomen	wood	lead	traces of plaster	Main late Roman Site E	Textile impressions	PD892 dark residues adhering to plaster with textile impressions
Grave 922	50	M	standard	wood	---	plaster in fill *calcite (lime)	Main late Roman Site E	Textile impressions	PD922 dark residues adhering to plaster with textile impressions
Grave 1040	30	M	standard	wood	lead	body-casing *lead substituted carbonate	Main late Roman Site E	Textile impressions	PD1040 dark residue adhering to plaster with textile impressions
Crown Buildings; Accession Number 1998.38; SY 6870 9068; Sparey Green *et al.* 1982									
	c. 25	M	extended healed injuries	wood	lead	body-casing gypsum		Textile fragments, plain weave linen ?shroud Hair preserved, coated in 'black tarry' substance	CGB H1 matted hair fragments CGB H2 end portion of plait CGB V associated material
Alington Avenue, Fordington; Accession Number 1991.89; SY 7024 8995; Davies *et al.* 2002									
G3664 SF 1075	>45	F	extended head to SW	wood sandstone lid	---	Reported as packed with plaster *chalk rubble from burial environment	Within enclosed area	3rd century AD Hobnails and leather, x2 pairs of footwear, 1 worn Glass and pewter vessels at head end of burial Black burnished ware jar above lid Fish bones, ?food offering	AA3664c grave deposits, head end AA3664f residue on foot bone
G4378 SF 1169	4-6	?	extended head to SE	wood	lead	Reported as a plaster burial *calcium carbonate inwash from the burial environment	Within enclosed area	3rd century AD Textiles adhering to upper torso comprising wool with purple-dyed decorative stripes (*clavi*) Black burnished ware jar *As* of Marcus Aurelius Iron rod	AA1 C1 grave deposits, head end AA3m residue, mandible AA4r residue, rib bones AA5cv debris, cervical vertebrae AA6 C2 grave deposits, head end AA7 h residue and debris, humerus

7.8.3.2 Site details and results: Crown Buildings

In 1971, a small burial ground was encountered during re-development work on the site of Crown Buildings outside the west gate of Dorchester. Situated between the roads to Ilchester and Exeter, around fifty graves were identified. The majority contained extended inhumations in wooden coffins orientated with head to the west without extant grave goods, suggesting a late Roman date. At least one individual had, however, been interred in a lead-lined coffin with cable moulding (Sparey Green *et al.* 1982). These skeletal remains were found to comprise those of an adult male, c. 25 years old, and showed evidence of a number of minor, healed, injuries (Harman 1982). He had been wrapped in plain weave linen, probably the remains of a textile shroud and encased in gypsum (Crowfoot 1982; Freeman 1982). A significant quantity of hair was also recovered which, upon examination, proved to be a 'head' of plaited reddish-brown human hair and a 'pig-tail', probably from the same individual (Paterson 1982; **Figure 7.48a**).

Figure 7.48. Human hair, Roman period plaster burial, lead-lined coffin, Crown Buildings, Dorchester, Dorset, UK: a. plait showing oily residue and traces of plaster; b. solvent extraction of lipids (Author). Scale bar: 1 cm.

Three samples were obtained from this burial. These consisted of two solvent washes of the hair (loose fragments and end portion of the plait) which had an oily appearance (**Figure 7.48b**) and one of associated orange-coloured debris. The hair washes contained only traces of saturated ($C_{16:0}$ and $C_{18:0}$) and unsaturated ($C_{18:1}$) carboxylic acids, generic indicators for the presence of a fat or oil. The sample recorded on the plastic bag by the excavators as 'vegetable matter' showed the presence of traces of ubiquitous lipid species which consisted of *n*-alkanes, *n*-alkanols and a series of SFAs with $C_{16:0}$ as the dominant compound. These are end-products of the decomposition of many organic substances (plant and animal) and cannot be assigned a specific source.

7.8.3.3 Site details and results: Poundbury

Throughout the 20th century AD, building work to the north-west of Dorchester repeatedly revealed evidence of a Roman burial ground in the area known as Poundbury/Poundbury Camp, now the Grove Trading Estate (Farwell 1993). Excavations carried out in the 1960s-1970s uncovered a long sequence of settlement activity (Sparey Green 1987). During the 1st century AD, a PRIA burial ground (Site C) was also established with crouched inhumation (c. 60 graves recovered) extending into the early Roman period. Subsequent developments on Site C were accompanied by extension of the burial ground to include the main late Roman cemetery (4th century AD) and peripheral burial groups. A small number of cremation burials and over 1300 inhumation burials were recovered from these areas with the vast majority interred supine, extended, in wooden coffins and accompanied by hobnailed footwear but with little in the way of extant grave goods (Farwell 1993). Those from the main burial ground were predominantly orientated head to the west and aligned in rows. Hence, the possibility that this was a Christian cemetery has been posited (Sparey Green 1982; Woodward 1993).

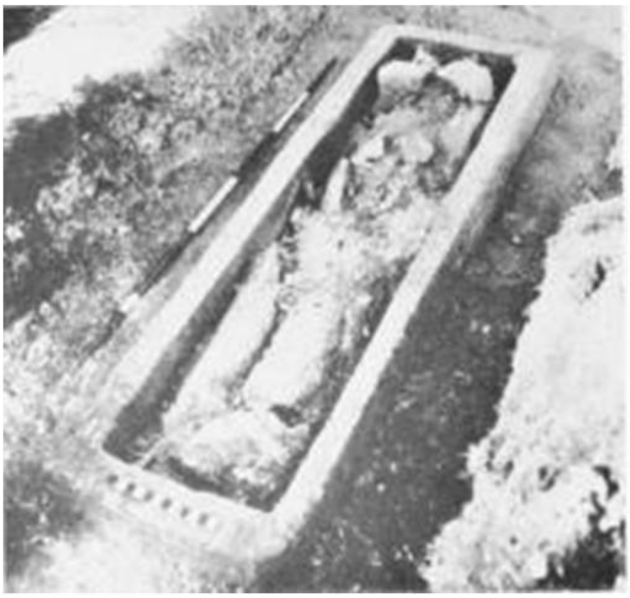


Figure 7.49. Plaster burial in sarcophagus, main late Roman burial ground, Poundbury, Dorchester, Dorset, UK (Image from archives, DCM, Dorchester ©DMT).

In addition, more elaborate mortuary treatments were observed within the late Roman burial area including interments in stone sarcophagi or lead-lined wooden coffins (**Figure 7.49**). Many of these appeared to be associated with ditched funerary enclosures or stone-built mausolea (Farwell 1993). A considerable number of these individuals (#31) had been encased in hydrated lime or gypsum with some standard wooden coffin burials also found to have included amounts of plaster (Davies and Grieves 1987; Woodward 1993). This treatment of the body may have contributed to the survival of hair which was recovered from a number of burials but it did not favour long-term skeletal preservation (Molleson 1993: 205-206). Likewise, fragments of textiles, mineral-replaced textiles (**Figure 7.50a**) and textile impressions (sometimes with associated staining; **Figure 7.50b**), indicative of the use of shrouds, were observed in many of these graves (Davies and Grieves 1987). Other finds included a ‘pillow’ of leaves below the cranium of a mature adult female (Grave 529; Keen 1979), a dyed woollen hair band with the remains of an adult male (Grave 530) and a tangle of gold thread by the left foot of a mature adult male (Grave 99; Crowfoot 1993; **Figure 7.50c**).

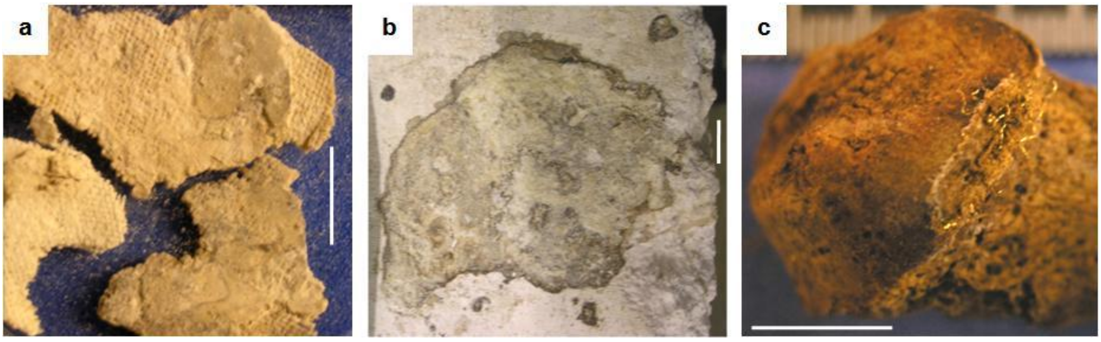


Figure 7.50. Materials recovered from burials, Poundbury, Dorchester, Dorset, UK: a. mineral-replaced textiles, Grave 127; b. textile impressions and staining, plaster, Grave 530; c. gold threads round head of first left metatarsal, Grave 99 (Author). Scale bars: 1cm.

In order to ascertain the nature of any compounds in the burial environment, a sample was taken from the outer surface of the plaster (identified as gypsum), from Grave 8. This showed that background values, in addition to phthalate plasticisers, consisted of a series of *n*-alkanes ($C_{16\text{-}28}$). Similar traces of *n*-alkanes were observed in the majority of the other samples. As these ubiquitous end products are non-diagnostic and could have derived from a wide range of sources, including the plastic wrappings, they will not be discussed further.

No volatile organic compounds (VOCs) associated with human decomposition or hydroxy- and oxo-carboxylic acids characteristic of adipocere were noted. A number of samples (#12) did, however, contain an homologous series of SFAs, generally eight to eighteen carbon atoms long, with either $C_{9:0}$ or $C_{16:0}$ dominant. The only unsaturated carboxylic acid recorded (in #8 samples) was octadecenoic acid ($C_{18:1}$). A range of *n*-alkanols was also found in conjunction with these acid moieties. In five samples (PD8 G2i; PD8 G5; PD517; PD529 and PD530a) these simple alcohols fell within the C_{10-18} range with a maximum either at C_{12} or C_{13} while in PD868, PD892 and PD1040 they ranged from C_{12-32} with a bimodal distribution (maxima at C_{12} and C_{28}). Traces of sterols (cholesterol; *β*-sitosterol) and 5α-cholestanol (in PD892) were also observed in the latter three samples. This combination of input sources is typical of soil organic matter and/or the microbial degradation of intrinsic deposits (e.g. human remains, floral offerings, degraded textiles).

Moreover, compounds characteristic of natural resins of archaeological interest were observed in nine samples from seven of the Poundbury burials (**Appendix 6.7**). Those identified were found to comprise diterpenoids and diterpenes with abietane and pimarane skeletons, biomarkers for members of the Pinaceae (**5.3.2**). In the sample obtained from the inner surface of the gypsum, Grave 8, a dark smear associated with textile impressions, this conifer resin was relatively well preserved as demonstrated by the predominance of the resin acids (PM, SDPM, IPM and AB) alongside a lower abundance of their derivatives (**Figure 7.51; Table 7.23**). Degradation products such as DHA, its oxidation artefact 7-oxodehydroabietic (7ODHA) acid and defunctionalised norabietatrienes and norabietatetraenes were, likewise, detected in Grave 892 and Grave 1040. The remainder (samples from Graves 127, 517, 529, 530) showed evidence only of more extensively altered neutral compounds including retene and/or methyl dehydroabietate. These highly resistant end products, alone, would not be sufficient to indicate an archaeological resin. In combination, however, these samples can be considered consistent with the presence of naturally aged Pinaceae products although those containing only the more degraded compounds could be indicative of thermal processing in antiquity.

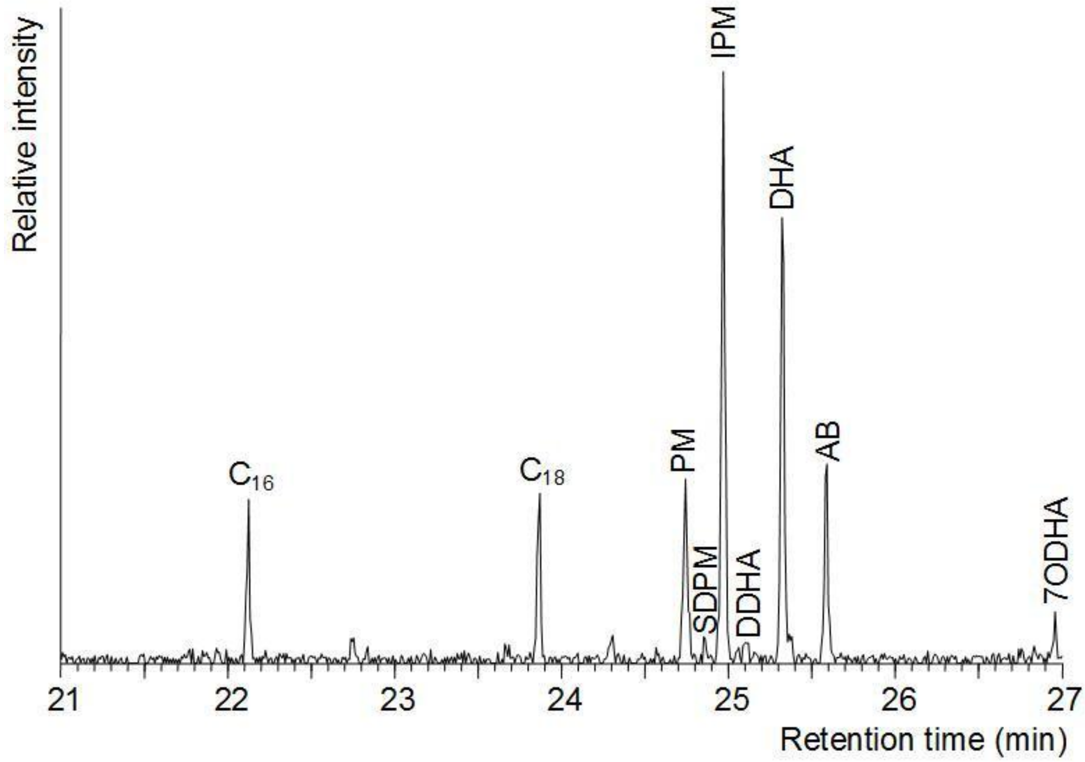


Figure 7.51. Partial XIC (*m/z* 241) diterpenoids, inner surface, gypsum, Grave 8, Poundbury, Dorchester, Dorset, UK. Peak identifiers relate to **Table 7.23**.

Table 7.23. Identification of diterpenoids (TMS derivatives), inner surface, gypsum, Grave 8, Poundbury, Dorchester based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	M^{+}•	BP	Key fragment ions	Name of compound
PM	374	73	121, 133, 191, 207, 257, 299, 359	Pimaric acid
SDPM	374	121	73, 91, 143, 241, 257, 359	Sandaracopimaric acid
IPM	374	241	73, 105, 143, 256, 257, 359	Isopimaric acid
DDHA	370	237	73, 103, 143, 195, 209, 252, 355	Didehydroabietic acid
DHA	372	239	73, 129, 143, 171/3, 185, 240, 255	Dehydroabietic acid
AB	374	256	73, 105, 185, 213, 241, 257	Abietic acid
7OHA	386	253	73, 143, 187, 268, 327	7-oxodehydroabietic acid

Confirmation of the botanical nature of this resin was made through comparison with modern Pinaceae products (**Appendix 3.1**). Evaluation of this data demonstrated that the mass spectra of the diterpenic compounds in the archaeological samples matched those in the modern materials. Those in the best preserved example from Grave 8 appear most similar to *Pinus* spp. exudates which are characterised by significant amounts of abietic acid (represented in PD8 G2i by AB, DHA, DDHA and 7ODHA) together with pimaric acid and its isomers. In addition, the relative abundance of MDHA and retene, indicators of thermal alteration, in the *P. sylvestris* tar are in contrast to their low levels in PD8 G2i indicating that the ancient resin had probably not undergone extensive, if any, heating prior to deposition.

7.8.3.4 Site details and results: Alington Avenue

In 1984-1987, excavations at Alington Avenue, Fordington (SY 702 899), to the south-east of Dorchester revealed over 100 inhumation burials and three cremation burials associated with a 1st-4th century AD farmstead. Those interred during the late Roman period (post c. 175 AD) were located within a small burial enclosure situated beside one of the approach roads to the Dorchester. Nine crouched inhumations (c. AD 50-125), reflecting PRIA traditions, were found outside the boundary of the enclosure, two with hobnailed footwear. Finds within the enclosure indicated that this area was in use c. AD 150-350 and contained three cremation burials in Black Burnished ware pots (mid-2nd-late 3rd century AD), ninety-one extended inhumations and a number of 'empty' graves. Most individuals were interred in simple nailed oak coffins with a range of grave goods. Hobnails, representing footwear (often worn), were particularly common and many had coins placed in or near the mouth. In addition, six infant burials were found nearby, below a late 3rd century building (Davies *et al.* 2002: 60-69, 122-146, 197-199).

Despite its small size and rural location, a number of significant inhumation burials were present. These included the normative burial (extended, supine, coffined) of an individual with Langer mesomelic dysplasia (SF 766), a form of dwarfism (Rogers 2002). In addition, two more elaborate 3rd century AD burials were recovered. The first, a mature adult female (SF 1075), had been interred in a wooden coffin with substantial stone lid (Grave 3664) and was reportedly packed in gypsum (**Figure 7.52a-c**). Leather and hobnails denoting the presence of footwear (x2 pairs) and an array of glass, ceramic and pewter vessels and some fish bones accompanied this individual (Davies *et al.* 2002: 131-139).

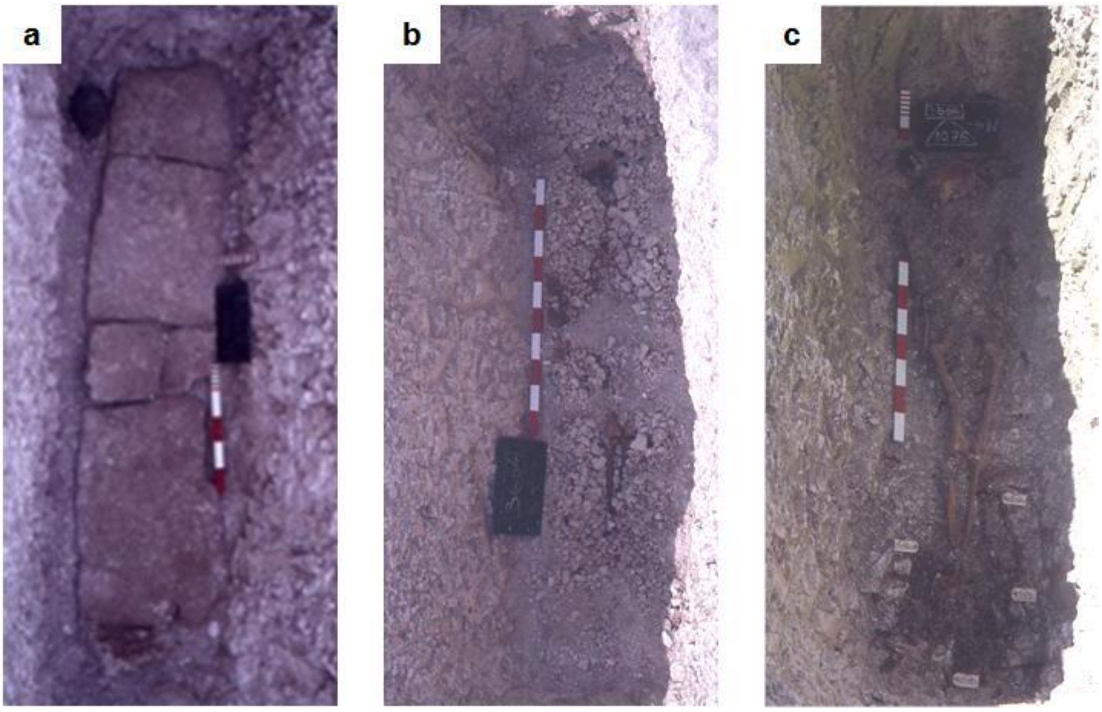


Figure 7.52. Alington Avenue, Fordington, Dorset, UK, Grave 3664 during excavation: a. stone slab used as a lid; b. skeleton 1075 and rubble infill; c. skeleton after infill removed, position of hobnails marked (Images from archives, DCM, Dorchester ©DMT).

The second, comprised the remains of a child aged around 4-6 years old (SF 1169) who had been placed in a lead-lined wooden coffin (Grave 4378) furnished with a Black Burnished ware jar, iron rod and curated coin of Marcus Aurelius (**Figure 7.53a**). The latter burial was intact and well preserved resulting in the survival of textile fragments adhering to the clavicles and scapulae (Davies *et al.* 2002: 133-135). When analysed, these proved to be a wool plain-weave (tabby), probably undyed, with weft-faced sections dyed with shellfish (murex) purple. These probably derived from a traditional form of Roman garment manufactured in the Mediterranean or Levant. The first of its kind to be discovered in Britain, such tunics were largely reserved for senators, civilian officials, army officers and young boys of high rank, presumably the latter in this instance (Crowfoot 2002; Walton Rogers 2002). The inhumation of an infant, c. 6-16 months old (SF 245) in a wooden coffin accompanied by an high quality glass vessel and located within a substantial grave cut, possibly below a wooden chamber, was also noted (Davies *et al.* 2002: 135).

A number of samples were obtained from Grave 4378, the lead-lined coffin of the child. These comprised dark residues and associated debris from the mandible (AA3M), rib bones (AA4R), cervical vertebra (AA4CV) and upper humerus (AA7H). Two samples were also collected from a bag containing unprocessed grave deposits from the base of the coffin (AA1C1; AA6C2). Analysis provided evidence of phthalate plasticiser contaminants and a range of *n*-alkanes (C_{15-29}), *n*-alkanols (C_{20-30}, C_{28} max.) and SFAs ($C_{14:0,\ 16:0,\ 18:0}$), generic end products denoting degraded plant and/or animal matter. In addition, a series of triterpenic compounds was observed.

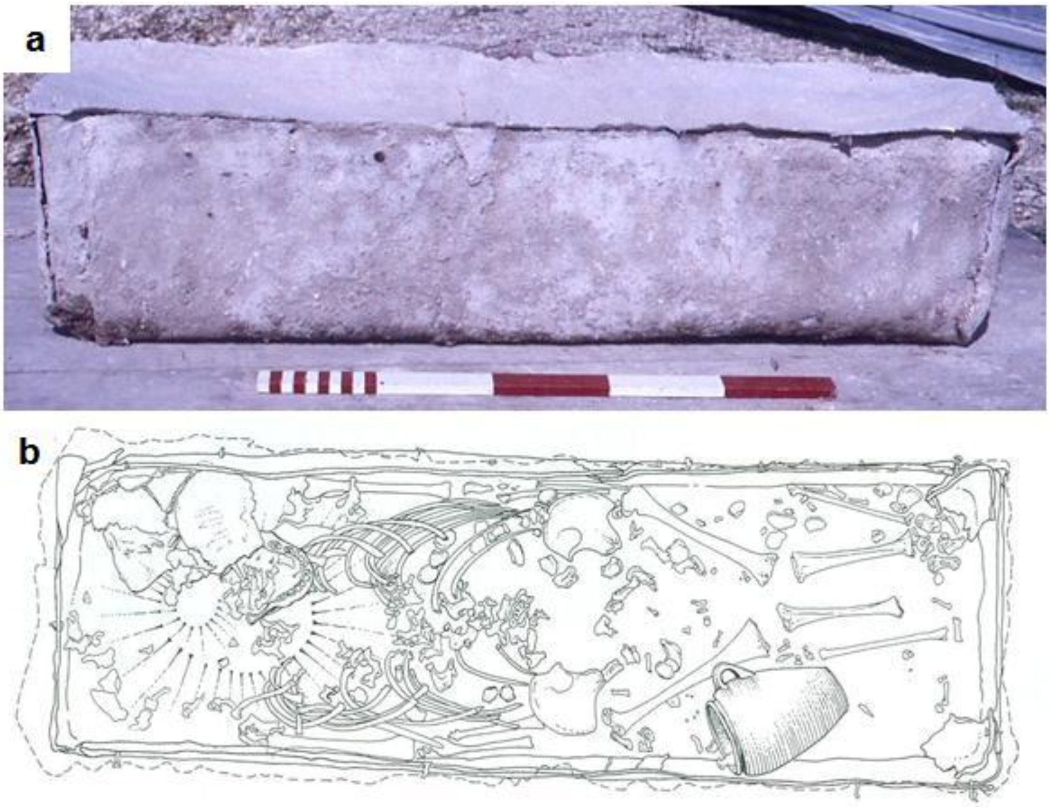


Figure 7.53. Alington Avenue, Fordington, Dorset, UK, Grave 4378: a. intact lead-liner after lifting (Image from archives, DCM, Dorchester ©DMT); b. scale drawing of skeleton 1075 and associated grave goods (Figure from Davies *et al.* 2002: Figure 6.3, 141).

As a subsequent visit to Dorchester had been planned to review the paper and photographic archive, further sub-samples of the grave deposits from the proximal (AA4378H) and distal (AA4378F) ends of the lead-liner were collected to confirm these findings. Although the liner was intact, it should be noted that a loose tooth was found in the bag from the foot region which indicated that some disturbance had occurred within the container, probably as a result of water ingress (denoted by white 'tide marks' which proved to be calcite from the surrounding burial environment with lead substitution; Schotsmans 2013: 195) as the remains were largely in anatomical alignment (**Figure 7.53b**). Likewise, materials from within the bags containing the cleaned skeletal remains from Grave 3664, the adult female in the wooden coffin with stone lid, were assessed. Although the residue from the foot bones (AA2F) was insufficient for analysis, the sample associated with the cranium (AA3664C) contained a near identical range of compounds to those obtained from Grave 4378. Again, although reported as a plaster/gypsum burial, analysis showed that the rubble infill comprised chalk from the burial environment (Schotsmans 2013: 195; **Figure 7.52b**).

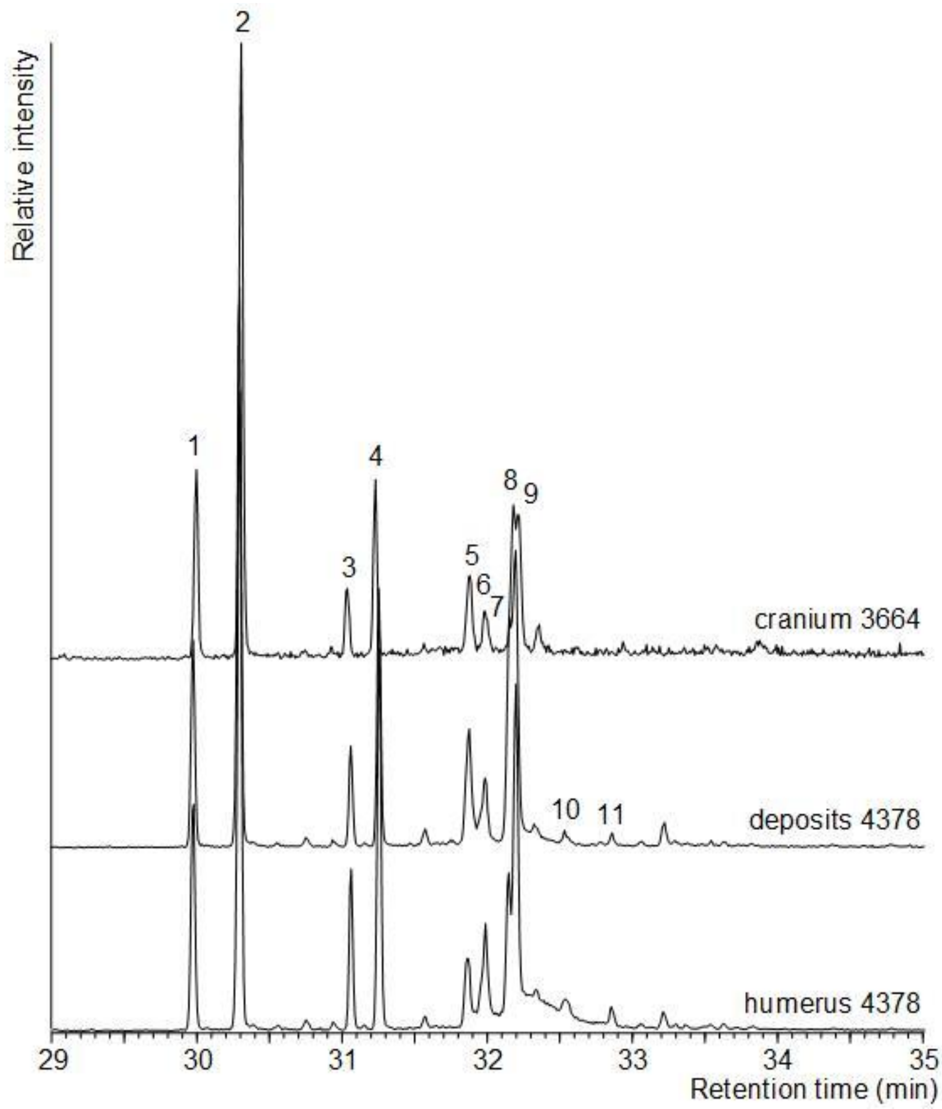


Figure 7.54. Partial XIC (*m/z* 218) triterpenic compounds, Graves 3664 and 4378, Alington Avenue, Fordington, Dorchester, Dorset, UK. Peak identifiers relate to **Table 7.24**.

In both cases, the pentacyclic triterpenic compounds identified were found to have oleanane and ursane skeletons based on their characteristic fragmentation patterns (**Figure 7.54; Table 7.24**). Significant peaks at *m/z* 189, 203 and 218 are characteristic of olean-12-ene and urs-12-ene derivatives and, alongside a greater abundance of the latter, are diagnostic of angiosperm resins from the Burseraceae family (Başar 2005: 117-119; Stacey *et al.* 2006). Moreover, the presence of the α- and β-boswellic acids and their degradation products, 24-noroleana-3,12-diene, 24-norursa-3,12-diene and 24-norursa-3,12-dien-11-one (produced by decarboxylation and dehydration or deacetylation of the resin acids), alongside 3-*epi*-amyrins allowed identification to the level of genus. Distinguished by a methyl group at C-17, which results in a base peak at *m/z* 218, these compounds are biomarkers for *Boswellia* spp. gum-resins, better known as frankincense (Budzikiewicz *et al.* 1963; Modugno *et al.* 2006b).

Table 7.24. Identification of triterpenic compounds (TMS derivatives), Graves 3664 and 4378, Alington Avenue, Fordington, Dorset, UK based on molecular ion ($M^{+\bullet}$), base peak (BP) and key fragment ions.

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
1	394	218	379, 323, 257, 229, 203>189, 175, 161, 147, 135, 119	24-norolean-3,12-diene
2	394	218	379, 341, 281, 203<189, 175, 161, 147, 133, 119, 107	24-norursa-3,12-diene
3	498	218	393, 327, 279, 257, 203>189/190, 175, 147, 121	3-*epi*-β-amyrin
4	498	218	483, 408, 393, 229, 203<189/ 190, 175, 161, 147, 121	3-*epi*-α-amyrin
5	408	232	393, 353, 273, 255, 161, 135	24-norursa-3,12-dien-11-one
6	424	218	409, 391, 367, 313, 257, 203, 189, 175, 161, 135, 109	β-amyrenone
7	498	218	483, 468, 408, 393, 311, 241, 203, 189, 161, 129, 69	β-amyrin
8	424	218	409, 393, 311, 257, 245, 203, 189, 175, 161, 135, 121	α-amyrenone
9	498	218	483, 468, 408, 393, 279, 257, 203=189, 175, 135, 119	α-amyrin
10	600	218	585, 510, 495, 382, 292, 203, 189, 161, 147, 135, 107	α-boswellic acid
11	600	218	585, 510, 495, 382, 292, 203, 189, 161, 147, 133	β-boswellic acid

Confirmation of this finding was sought through comparison with modern botanically and geographically identified *Boswellia* spp. resins: *B. serrata*, Sudan; *B. carterii*, Ethiopia and *B. sacra*, Oman obtained from Bristol Botanicals Ltd. Evaluation of the key triterpenic region demonstrated that compounds with identical fragmentation patterns were present in both the modern and archaeological samples with the ursane skeleton moieties consistently more prominent than their corresponding oleanane skeleton isomers (**Appendix 3.3**). No triterpenoids or their derivatives with lupane-based structures were identified in either the modern or archaeological samples. These have been reported in the literature as principally present in *B. frereana*, from Somalia (c.f. Culioli *et al.* 2003; Mathe *et al.* 2004b).

A range of diterpenes (cembrene isomers, verticilla-4(20),7,11-triene, incensol and derivatives) were also observed in the modern gum-resins from *B. carterii* and *B. serrata* with minimal evidence (possible trace of incensol) in *B. sacra* (**Figure 7.55; Appendix 3.3**). These compounds have been identified, although they varied in their presence/absence and relative abundance, in the exudates of most members of the genus *Boswellia* with the exception of *B. frereana* (Baeten *et al.* 2014; Başar 2005: 42-81; Hamm *et al.* 2003, 2005; **Table 7.25**).

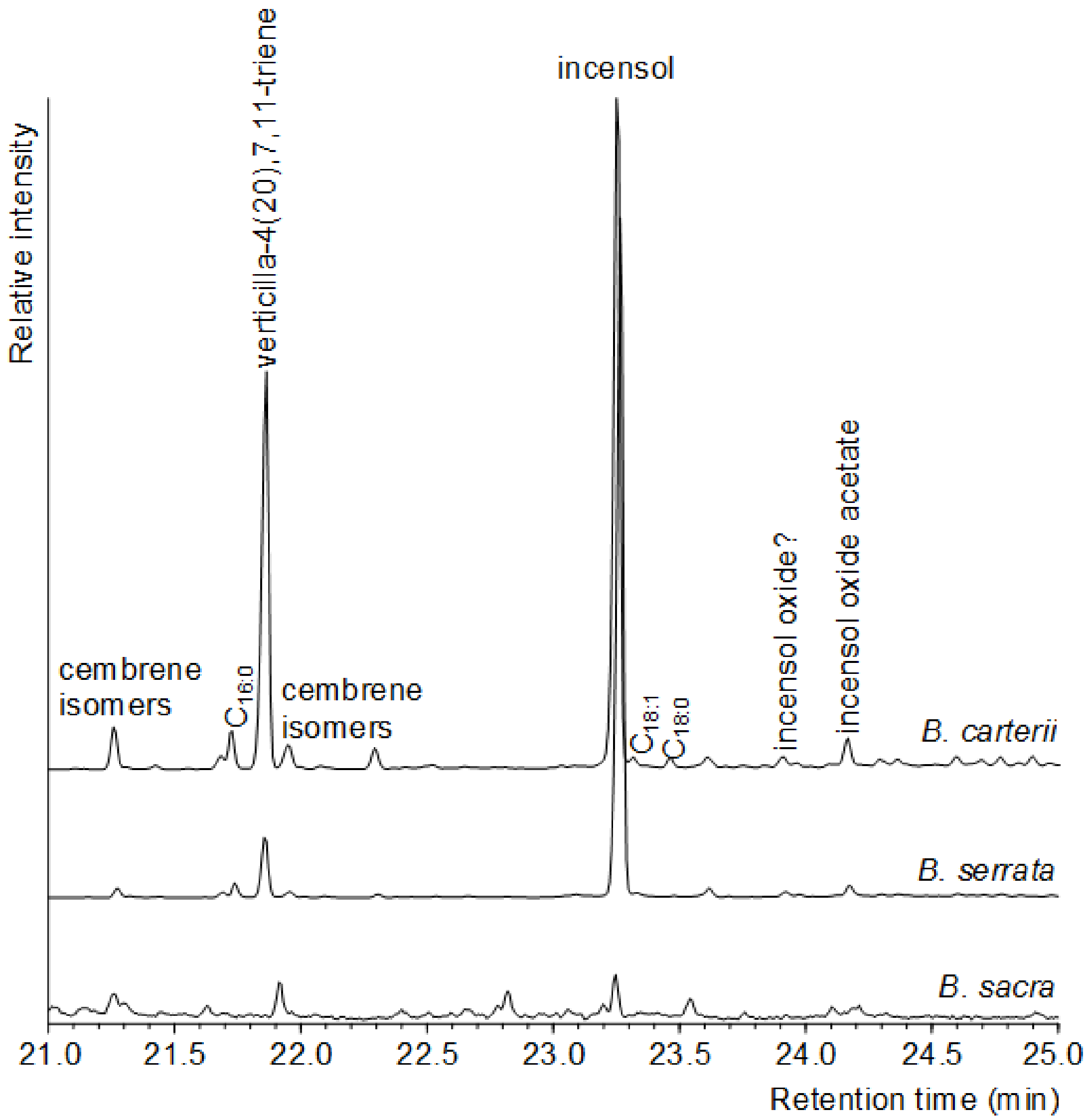


Figure 7.55. Partial TIC (21-25 min) diterpenic compounds, modern *Boswellia* spp. gum-resins, *B. carterii* (BB050), *B. serrata* (BB009) and *B. sacra* (BB052).

Table 7.25. Diterpenic compounds reported in *Boswellia* spp. gum-resins a. Baeten *et al.* 2014; b. Başar 2005: 42-81; c. Hamm *et al.* 2005. Percentage abundance indicated by: X <1.0; XX 1.0-4.99; XXX 5.00-9.99; XXXX >10.00.

***Boswellia* spp.**	***B. carterii***			***B. sacra***		***B. serrata***			***B. papyrifera***	
References	a	b	c	a	c	a	b	c	a	c
Cembrenes, generally A + C	XX	XX	X	XX	X	XX	X	XX	XX	XX
Verticilla-4(20),7,11-triene		XXX	X		X			X	XXX	
Incensol	XXXX	XX		X		X			XXX	X
*Iso*incensol or serratol/ *iso*incensol acetate	XXX	XX	XX	XXXX	XX	XXXX		XXX		
Incensol acetate									XXXX	XXXX
Incensol oxide/oxide acetate									X	X

Traces of these diterpenic compounds may be present in the poorly resolved 'hump' (20-25 min) in the Alington Avenue samples but none could be securely identified (**Figure 7.56**). In addition, it was noted that only partial silylation of incensol had occurred, presumably hindered by the complex conformation of this compound (**Figure 5.7**). The same finding has recently been reported by Baeten *et al.* (2014).

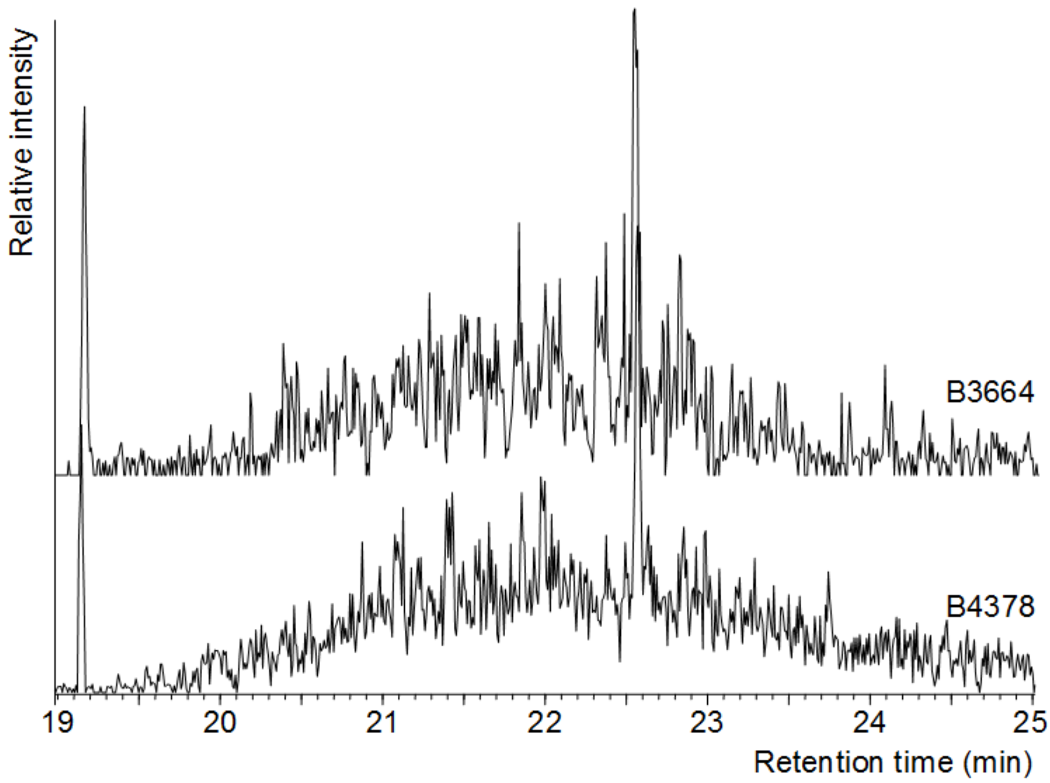


Figure 7.56. Partial XIC (*m/z* 272) possible traces of diterpenic compounds, archaeological samples, Graves 3664 and 4378, Alington Avenue, Fordington, Dorchester, Dorset, UK.

Despite this clear correspondence with *Boswellia* spp. gum-resins, the range of compounds and their relative abundances varied considerably between the modern and archaeological samples. The fresh exudates were dominated by the boswellic acids and their *O*-acetyl derivatives. In contrast, only trace amounts of the diagnostic resin acids were observed in the archaeological samples and their *O*-acetyl oxidation products were absent, replaced by an abundance of norterpenic derivatives. This pattern resembles the reduction in functionalised moieties and increase in neutral compounds reported as a result of pyrolysis, although nortrienes, thought to be markers of extensive heating (i.e. burning as incense), were not present in the archaeological samples (c.f. Başar 2005: 151-184; van Bergen *et al.* 1997; **Table 5.6**). More research is, however, needed to ascertain if environmental factors could result in similar diagenetic changes.

7.8.4 Summary and interpretation of findings

Analysis of materials recovered from Roman period inhumations from the environs of Dorchester, Dorset, UK demonstrated the presence of two different plant exudates. Organic residues associated with textile impressions on plaster body-casings of seven inhumations at Poundbury, showed a range of diterpenic compounds characteristic of coniferous Pinaceae exudates. Although most of these finds comprised only trace amounts of degraded abietic acid derivatives, the range of compounds in a more extensive residual layer from Grave 8 indicated the use of an unheated, possibly *Pinus* spp. resin. In addition, grave deposits and residues adhering to the skeletal elements of two individuals interred in a rural burial ground at Alington Avenue revealed biomarkers denoting the inclusion of frankincense (*Boswellia* spp. gum-resins). The heavily degraded nature of this exotic substance could be due to thermal alteration prior to deposition although, as more information is required regarding the impact of natural taphonomic factors, interactions over time within the burial environment might also account for its condition.

7.9 Case Study 8: Recent find near Ilchester, Somerset

7.9.1 Background

Towards the end of 2013, metal-detectorists discovered a lead-lined coffin in a field outside Ilchester, Somerset. The nature of the find was only ascertained after a pick-axe had penetrated the lower left portion of the lid and the distal end had been raised and re-lowered. This allowed ingress of some chaff and grains from the overlying arable field which were observed on top of the soil infill. Realising the significance of their find, the detectorists contacted the authorities and a rescue excavation was carried out under the supervision of Robert Croft, Somerset County Archaeologist. An area around the lead coffin was excavated and the find was lifted whole for micro-excavation and sampling in a laboratory setting.

This subsequent work revealed that the lead-liner tapered towards the distal end and had suffered considerable damage (**Figure 7.57**). The lid, a flat sheet of lead with an irregular lip folded over like a pie-crust (Type 2, Toller 1977: 11), had remained largely intact although twisted by mechanical disturbance and bowed by the soil overburden with parts of the lip severed by being pressed down onto the sides. The container itself had also suffered compression which had resulted in separation at the corners (no evidence of solder was observed) with the distal end and the left vertical panel, in particular, folded inwards over the body. The sides had been compressed medially and were indented in the knee area with large tears along the base on both sides. This severe disturbance, probably been caused by farm machinery (i.e. ploughing), had resulted in the extensive ingress of soil which almost completely filled the liner and covered the human remains. Only fragments of the cranial vault which were pressed into the top right corner and the line of the thoracic vertebrae could be detected once the lid had been lifted (**Figure 7.58**).

Removal of the soil in gridded spits (upper layer, A101-309; lower layer B101-309, in line with the ideal sampling strategy, see **6.2**) enabled the position of the skeletal elements to be recorded in detail and considered using the '*anthropologie de terrain'* approach devised by Henry Duday (2009: 3-14). Thus, as work progressed it became clear that this was a primary inhumation of a single individual who had been interred supine, extended with arms at the sides. The majority of the skeletal elements were recovered and had remained in rough anatomical order although some of the smaller bones (e.g. the patellae and bones of the hands and feet) had moved around the coffin. This is not uncommon and probably occurred when the lead-liner was largely intact as they were scattered across its base beneath the soil infill indicating that soft tissue decay had occurred in a void (c.f. Duday 2009: 32-57).

Figure 7.57. The lead-liner, Ilchester, Somerset in the laboratory prior to the lifting of the lid: a. right side; b. left side (Author).

Figure 7.58. Anterior surfaces of thoracic vertebrae, lead-lined coffin burial, Ilchester, Somerset revealed after the lead lid had been removed (Author).

The movement of these elements could have resulted from water/faunal action or from the container being struck by farm machinery. As a consequence, the cranium had fallen to one side and had subsequently been forced into the right corner of the liner and fragmented. The mandible lay over the upper cervical vertebrae so had become detached, naturally, before the movement of the cranium (**Figure 7.59**), as is observed during decomposition in a void (Duday 2009: 17-19).

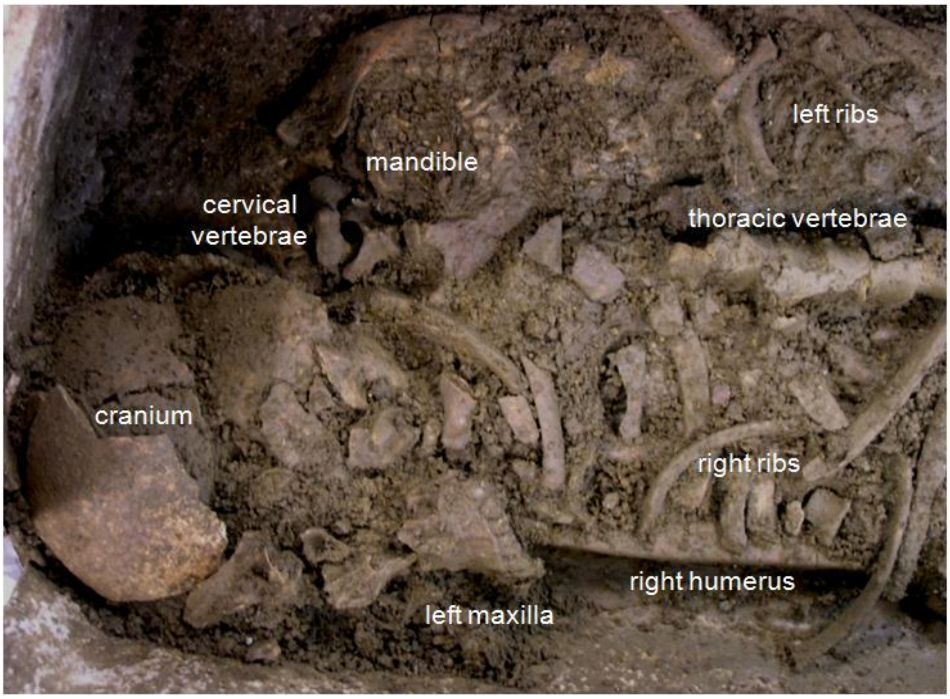


Figure 7.59. Cranium, mandible and upper torso, lead-lined coffin burial, Ilchester, Somerset during excavation in the laboratory (Author).

The arm bones remained fully articulated with the exception of the right radius which had become dislodged and lay horizontally over the body. Likewise, the os coxae and femora were still in anatomical association but the medial crushing had separated the legs at the knees, forced the sacrum and lower limbs towards the foot end of the coffin (distally) and towards the right hand side and had rolled the left tibia through 180° (**Figure 7.60**).

Initial examination prior to full cleaning of the remains indicated that, despite the damage, skeletal preservation was moderate-good and that these were the remains of a gracile, young adult female, dubbed the ‘Lady in Lead’. This was confirmed by subsequent analysis (Hopkinson 2014). No plaster was detected within the lead-liner nor was there any evidence of a shroud, garments or residues (other than soil) adhering to the bones.

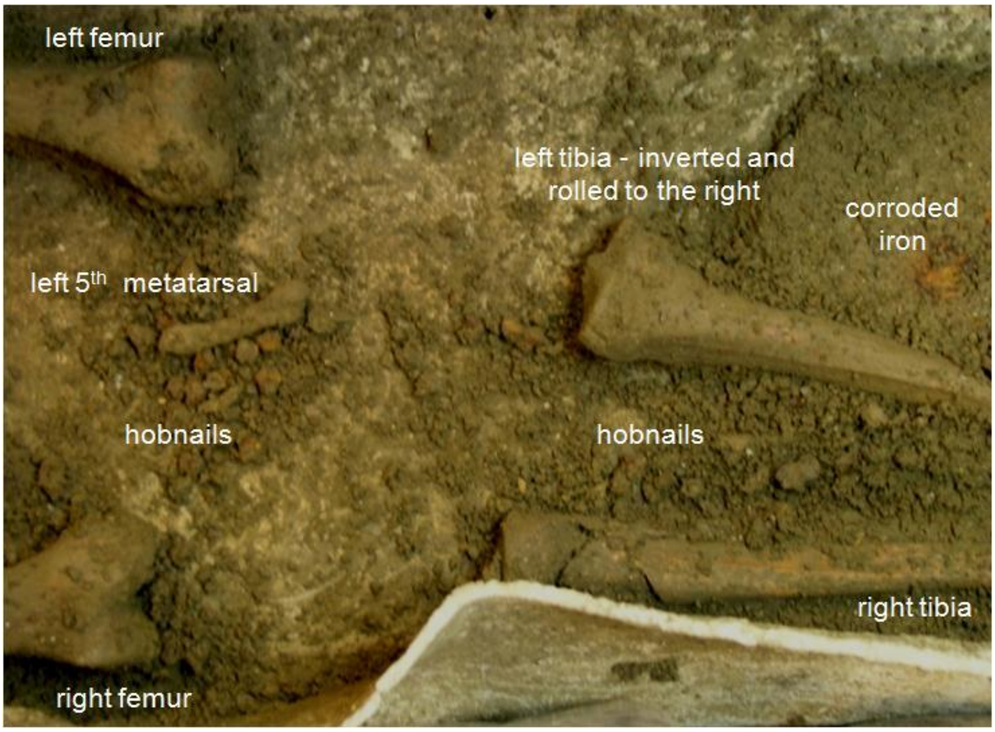


Figure 7.60. Distal femora and proximal tibiae lead-lined coffin burial, Ilchester, Somerset during excavation in the laboratory showing hobnails and left metatarsal (Author).

The only accompanying items to survive were two areas of hobnails denoting footwear. The remains of one of these ‘shoes’ was located at the foot of the coffin while the other was somewhat more scattered between the knees. There is considerable debate as to whether or not such footwear was worn at the time of burial or included to facilitate the journey. Due to the disturbance within the lead-liner this question cannot be definitely determined. However, as the left 5th metatarsal was found in association with the hobnails that appear to have migrated proximally, it seems that the footwear was probably worn in this instance and that, after soft tissue decomposition, this bone, perhaps trapped by an outer seam had been transported with the shoe. Wet sieving of the soils (after collection of samples for residue analysis) revealed a range of micro faunal remains and a cylindrical bead, possibly made of jet, with incised decoration.

7.9.2 Sample selection

Thirteen soil samples (~5 g) were collected from around the skeletal remains (from basal grid B) in order to gain an insight into the distribution of any lipids of archaeological relevance present. One control sample (SSCN) was also selected from the soil above the body (A104) to ascertain background environmental components (**Figure 7.61**).

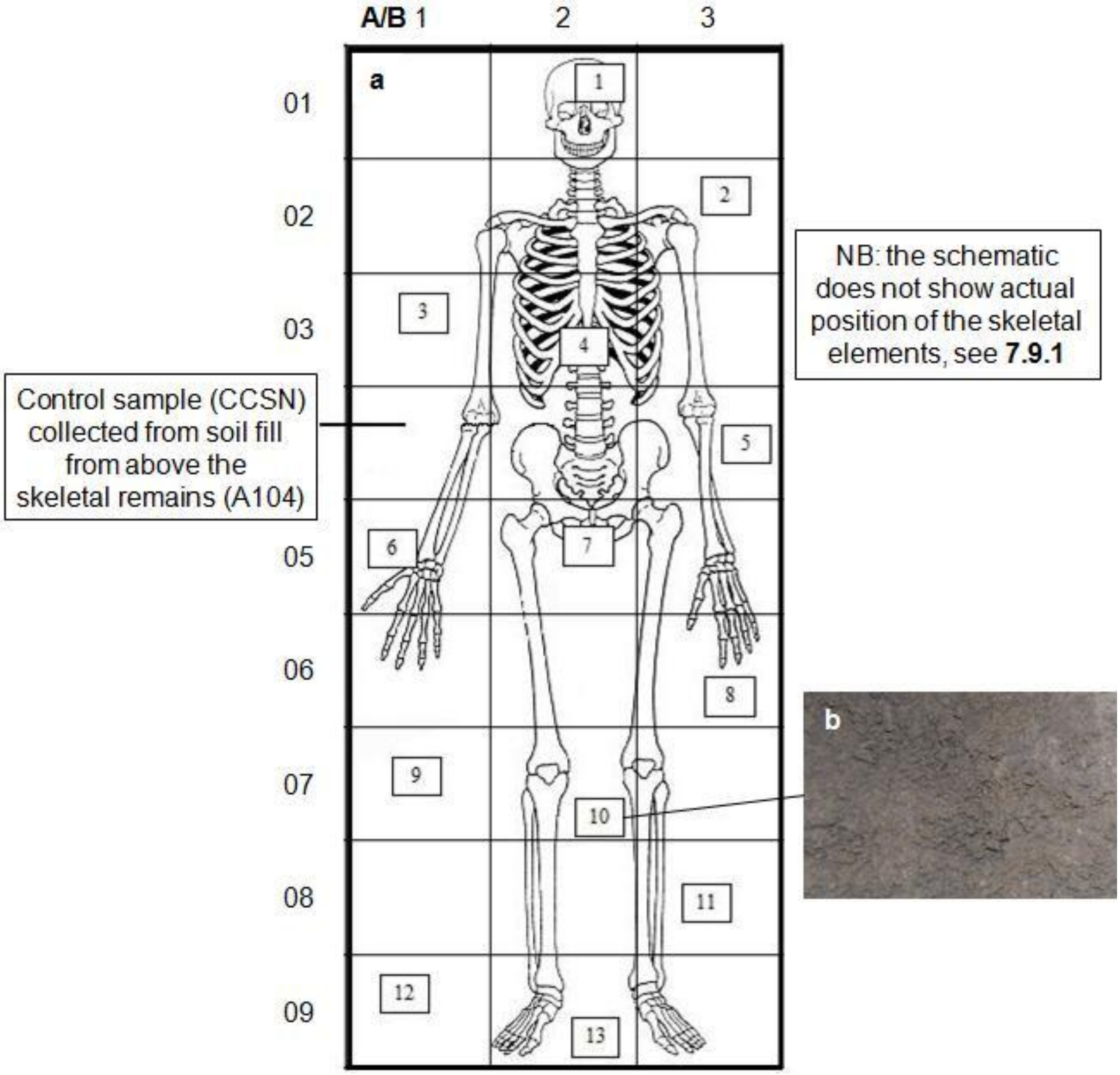


Figure 7.61. Diagram showing grid system employed during excavation of the lead-lined coffin burial, Ilchester, Somerset: a. position of soil samples collected from lower B layer and of control sample selected for analysis (numbers relate to GC-MS identifier, see **Appendix 6.8**); b. area between distal left femur and proximal left tibia sampled (SS10) (Author).

7.9.3 Results

The control sample (SSCN) contained a range of lipid species. These comprised *n*-alkanes ($C_{21-33,}$ C_{31} max), a significant abundance of *n*-alkanols ($C_{20-36,}$ C_{26} max), traces of SFAs ($C_{14:0-28:0}$, bimodal, $C_{16:0/26:0}$ max) including branched chain moieties (C_{15-17}), MUFAs ($C_{16:1}$; $C_{18:1}$) and steroidal compounds (cholesterol; 5α-cholestanol; campesterol; stigmasterol; *β*-sitosterol; stigmastanol). Likewise, all of the samples collected from the basal layer of soil associated with the human skeletal remains (SS1-SS13) were found to contain a near identical suite of components (**Figure 7.62;** for TICs and details by sample, see **Appendix 6.8; Disc 1, File 6.8**).

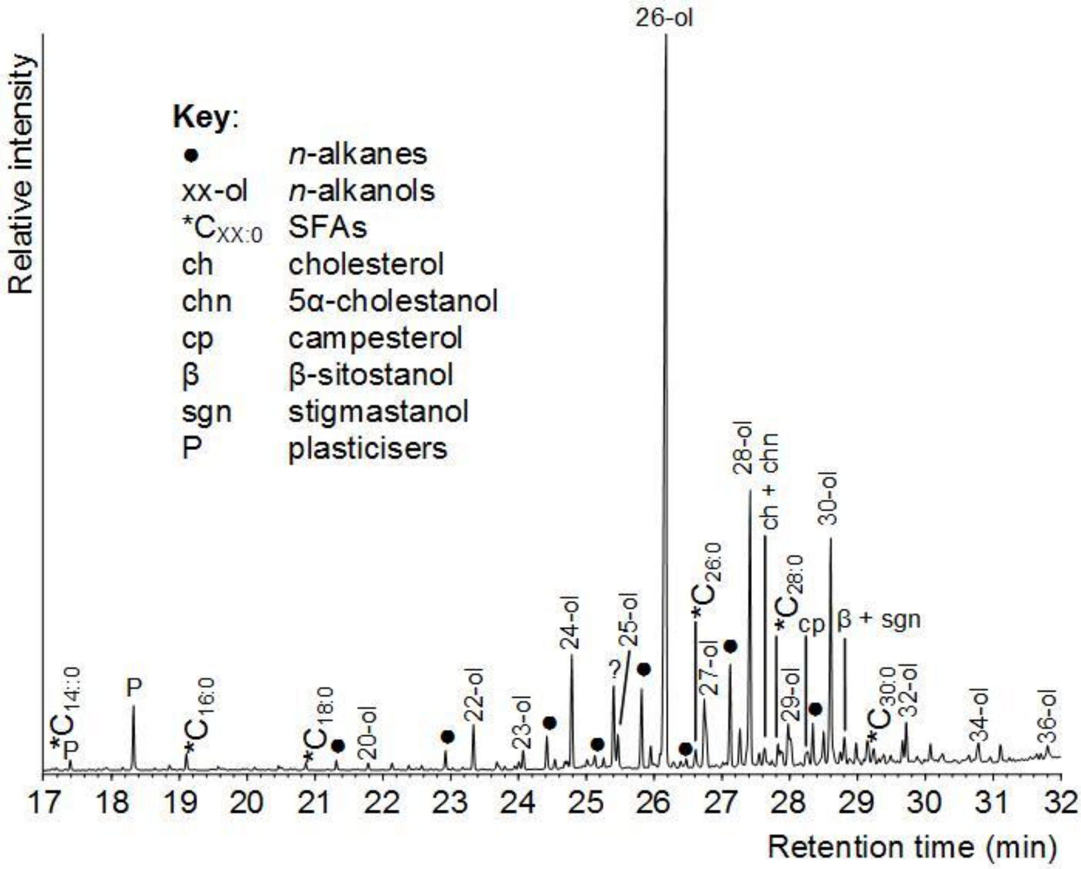


Figure 7.62. Partial TIC (17-32 min) sample lateral to left femur/near left hand bones (SS8/B306), lead-lined coffin burial, Ilchester, Somerset.

Thus, the majority of the lipid components throughout the burial were found to derive from degraded plant matter, probably cereal/grass roots, based on the range and relative abundances of various species present (Jambu *et al.* 1993; Otto and Simpson 2006). Some contribution from the decomposition of faunal remains (cholesterol; 5α-cholestanol) may also be indicated. These findings confirm the visual assessment made once the lid had been lifted that damage to the lead-liner had resulted in excessive soil ingress and that no evidence for treatment of the body was likely to remain. The range of compounds present is consistent with modern usage of the field in which the burial was discovered for arable farming.

7.9.4 Summary and interpretation of findings

It can, therefore, be concluded that the overlying vegetation was the main source of the lipids identified and that no residues (relating to body decomposition or treatment) of archaeological relevance remained. Likewise, no traces of textiles were recovered although footwear was clearly present (hobnails) indicating that considerable losses had occurred. Resinous substances may, of course, never have been present. Nonetheless, these negative findings from the only recently-discovered Roman period ‘package’ burial available for direct sampling by the author (in conjunction with similar issues encountered with a lead-lined coffin from Leicestershire, assessed by Prof. Keeley, University of York; 2013, pers. comm., 12-13 December) suggest that sampling for organic traces relating to the human remains, themselves, or to any body treatment is unlikely to prove successful where extensive disturbance (e.g. soil ingress and/or water movement), is evident.

7.10 Case Study 9: Plaster burials around York (Brettell *et al.* 2015a)

7.10.1 Background

York (*Eburacum*), North Yorkshire, UK (SE 600 519) was founded *de novo* around 71 AD during the campaigns of the Roman governor, *Cerialis*. This move to the north was aimed at establishing Roman control after a period of conflict between Cartimandua and her estranged husband Venutius, co-rulers of the client kingdom of the Brigantes (RCHME 1962: xxix). A network of forts was established in the region to maintain a military presence and included the construction of a timber legionary fortress by the IX *Hispania* on a raised plateau at the confluence of the rivers Foss and Ouse to act as an operational hub. Later rebuilt in stone (c. AD 107-108), this military base appears to have had a standard gridded layout, basilica, barracks and bathhouses and was rapidly integrated into the Roman road system becoming the main administrative centre for the region (Mattingly 2006: 256-257; Ottaway 2004: 23-49; RCHME 1962: 4-6). Evidence of substantial waterfront revetments and warehouses and craft areas with tile, pottery and glass working activities indicates that an economic centre (*canabae*) developed around the fortress (Ottaway 2004: 87-89). This then spread to the south-west bank of the Ouse with a separate civilian settlement, possibly a *municipium*, established in the late 1st century AD (Mattingly 2006: 192; RCHME 1962: 49-58).

In the early 2nd century AD, the IX *Hispania* was replaced by the VI legion, *Victrix*. This unit appears to have remained in York until the end of the Roman period (RCHME 1962: xxxii). During these centuries (2nd-4th AD), the continued prosperity of *Eburacum* was marked by the addition of colonnaded public buildings, a sewer system, fountains, bathhouses and private dwellings (town-houses) with hypocausts, courtyards, painted wall plaster and fine mosaic floors (Ottaway 2004: 134-136; Wacher 1974: 159-166). Finds of ceramics show that trade links extended across the Empire with Samian wares from Gaul, colour-coated vessels from the Rhineland and amphorae containing wine, olive oil and other exotic food-stuffs arriving from Italy, Spain and North Africa (Ottaway 2004: 103-107). Likewise, a number of villa complexes have been identified in its hinterland (Mattingly 2006: 420).

Such was the importance of York that, after the upheaval brought about by Albinus' bid for power, the fortress was reconstructed (c. AD 197) and given a more massive, turreted wall (RCHME 1962: 8-10). The city was then used by the emperor Septimius Severus as a base during his campaign against the peoples of Caledonia. Severus, who hailed from Tripolitania in North Africa and died in York in AD 211, was accompanied by an enormous entourage including his wife and sons. The remains of a substantial building near the river may be the site of the imperial palace (Southern 2013: 243, 253) although, as *Eburacum* become not only the capital of *Britannia Inferior* around this time but also an honorary *colonia* prior to AD 237, this building could be the governor's residence. This period of influence resulted in further remodelling of the military and civilian defensive circuits and, possibly, the layout of the town (Wacher 1974: 156-163; RCHME 1962: xxxvi-vii).

Little is known of the intervening period but in 305 AD, the emperor Constantius arrived in Britain having earlier (AD 297) restored the province to the Empire after its annexation under Carausius and Allectus (Southern

2013: 297-299). Like Severus, he not only chose York as the base for his northern campaigns but died there (AD 306) with his son, Constantine, declared successor in the city (Wacher 1974: 156). During the remainder of the 4th century AD, York appears to have maintained its status as the military headquarters of the north and capital of one of the new sub-divisions of the province created by Constantius (Wacher 1974: 161). There is also evidence for a slow decline with a reduction in imports, silting of the waterfront and the decay of the main bridge across the Ouse (Ramm 1971). Likewise, after c. AD 380 deterioration in the quality of repairs to the infrastructure (e.g. roads, sewers), the disuse of buildings and civilian occupation of the legionary fortress seems to have occurred (Mattingly 2006: 533-534; Ottaway 2004: 145-149; Wacher 1974: 176-177). Thus, as elsewhere in Britain, *Eburacum* effectively ceased to function in the early 5th century AD (Ramm 1971).

Following Roman law, a number of burial grounds existed outside the walls of York to serve the inhabitants of the fortress, *canabae* and civilian settlement (**Figure 7.63**). Predominantly located along the major approach roads, these have been found to incorporate a considerable diversity of mortuary practices from mass graves to elaborate vaulted tombs (RCHME 1962: 67-110). Unfortunately, most were discovered in the 18th-19th centuries AD so detailed records are scarce and little physical evidence survives (Ottaway 2004: 121). For example, of the twenty-seven lead coffin-liners from York discussed by Toller (1977: 41-42) only thirteen were still extant in the 1970s. Skeletal remains and any associated organic residues have fared even worse as the majority of individuals were re-buried. Nonetheless, a review of the main publications shows that over 100 stone sarcophagi, twenty-nine lead-liners and forty-nine plaster burials have been discovered around this important urban centre (Ramm 1971; RCHME 1962). This speaks to the significance of York during the late Roman period as London, to date, has produced only fifteen stone sarcophagi and fourteen lead-lined coffins although 115 plaster burials have been reported (**7.3**).

With the exception of the distinctive Arras culture of eastern Yorkshire, little is known about the treatment of the dead during the PRIA in northern Britain

(Whimster 1981). Post-conquest, however, the introduction of urned cremation as the standard form of burial in the more Romanised and militarised regions of Britain resulted in greater visibility of the dead in these areas (Philpott 1991: 8). Thus, in York the earliest evidence comes from small 1st-2nd century AD cremation cemeteries such as those at Fishergate, Burton Stone Lane and Heworth associated with the fortress and *canabae* (RCHME 1962: 69-70). During the 2nd century AD, as elsewhere in the Roman world, inhumation then began to supersede cremation although the latter continued as a minor rite (Philpott 1991: 54-55). This is best illustrated at Trentholme Drive where excavation revealed fifty-three cremations dated from around AD 160 and 342 inhumations spanning the late 2nd-early 4th centuries AD (Wenham 1968: 21-47). These finds formed part of the vast burial ground known as 'The Mount' (located on high ground along the road to the south-west of the *colonia*) from which, in conjunction with another major burial ground in the vicinity of the Railway Station, many of the more elaborate burials from York have been recovered (RCHME 1962: 76-106). Small groupings and isolated burials, however, stretch around the full circuit of both the city and the fortress (Ottaway 2004: 121-122; **Figure 7.63**).

In the later Roman period, the majority of individuals were interred supine, extended in wooden coffins although a considerable variety of other practices were employed. These included multiple inhumations, prone burials and decapitations with the use of a range of packing materials (e.g. stone, gravel, plaster) and burial containers (tile cists, stone sarcophagi, lead-lined coffins) (RCHME 1962: 67-110). Evidence for above ground mausolea and vaulted tombs has also been noted and a wealth of grave goods recovered (Ottaway 2004: 122-124; Wacher 1974: 171-176). Moreover, a considerable array of tombstones and funerary inscriptions provide details concerning the identities of those interred around *Eburacum* and those who chose to honour them. These memorials refer to soldiers, veterans, government officials and merchants who came from all over the Empire with their wives, children, freedmen and slaves (RCHME 1962: 111-135). Recently, osteological and isotope studies have provided support for this diversity of origins (Leach *et al.* 2009, 2010; Montgomery *et al.* 2011; Müldner *et al.* 2011). The beliefs of these individuals can, however, rarely be determined despite the diversity of deities attested (Mattingly 2006: 296-306; Wacher 1974: 165) although a number must have adopted Christianity in the 3rd-4th centuries AD as a bishop from York was present at the Council of Arles in AD 314 (Ottaway 2004: 137). The last burials appear to date from the mid-late 4th century AD with no evidence of continuity into the post-Roman period (Ramm 1971).

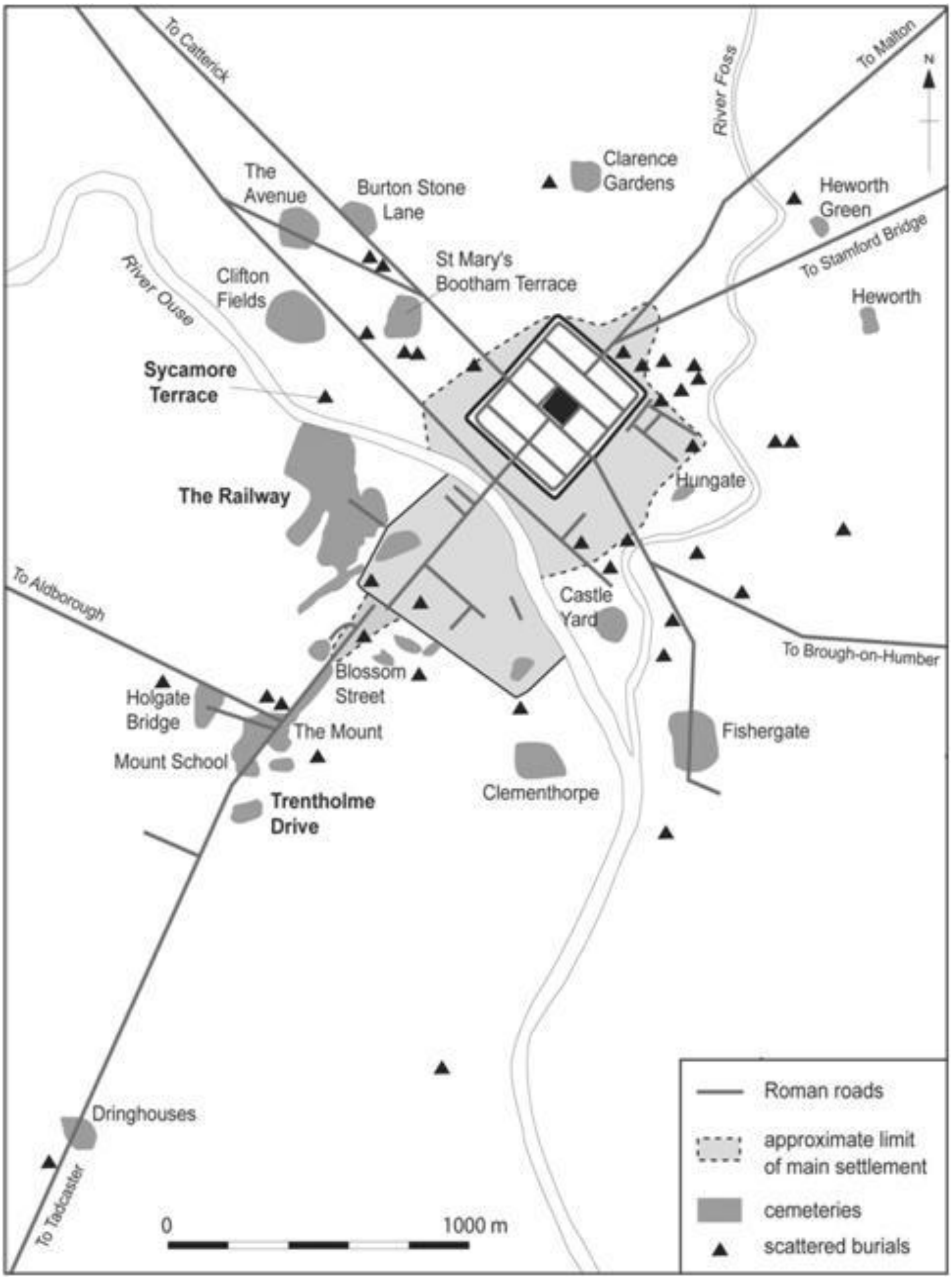


Figure 7.63. Location of the main burial grounds around Roman York (Figure modified from Müldner *et al.* 2011: Figure 1, 281).

Table 7.26. Inhumations from Roman period burial grounds around York, UK containing plaster assessed/sampled as part of this study.

Site	Identifiers	Details	Grave goods/fill	Records found	Samples collected
Ramm 1971; Royal Commission for Historic Monuments of England (RCHME) 1962					
York, unspecified **Probably:* *Railway excavations* *Found 1874*	YORYM: 2007.6207 Primary ref: 11932	Stone sarcophagus, particularly narrow *Reported as adult female	Plaster burial, cast above/around the body showing contours Textile impressions Grave goods: jet pin	*RCHME 1962: 85 (iv) Ramm 1971: 194 (33)	YK1: orange fragments/dark stained areas on plaster, cranial region YK2: orange fragments/dark stained areas on plaster, leg region
Railway excavations Found in 1848 SE side of present railway station	YORYM: 2007.6206 Primary ref: 11931	Stone cist 10 slabs of gritstone Flagstone floor Wooden coffin made of 'cedar wood'	Plaster burial, cast of body showing contours Textile impressions + fragments of coarse cloth	RCHME 1962: 84 (xiii) Ramm 1971: 194 (31)	YK3: yellow/dark stained areas on plaster, upper body region YK4: dark stained areas on plaster, near crack extending from outer surface YK5: yellow/dark stained areas on mineral-replaced textiles, cranial region
York, unspecified	YORYM: 2007.6208 Primary ref: 11933		Plaster burial, cast of body showing contours Textile impressions		YK6: orange residue embedded in plaster, leg region YK7: dark stained areas on plaster, leg region
York, unspecified	YORYM: 2007.6214 Primary ref: 11938		Plaster burial - lower body only, cast showing contours Textile impressions		YK8: dark stained areas on plaster, leg region
Mill Mount	YORYM: 2007.6205i Primary ref: 11928		Plaster burial, cast showing body contours, broken into x2 Textile impressions		YK9: dark stained areas on plaster, cranial region YK10: CONTROL, soil from crack in plaster
Railway excavations Found 1876	YORYM: 2013.152 Primary ref: 17802	Stone sarcophagus Infant 3^{rd}-4^{th} century AD	Plaster burial, cast of body showing contours Textile impressions	RCHME 1962: 83 (viii) Ramm 1971: 193 (27)	YK11: orange fragments + dark stained areas on plaster, cranial region
York, unspecified	YORYM: 2010.1201 Primary ref: 15425	Stone sarcophagus Inscribed	Plaster burial Hobnails + iron studs Some skeletal remains		YK12: brown debris with yellow flecks from base YK13: 2nd sample of debris YK14: scrapings from base of sarcophagus
Railway excavations Found 1892	YORYM: 2007.6212 Primary ref: 11936	Lead coffin Child 3^{rd}-4^{th} century AD	Plaster burial Textiles: 'linen cast', fine plain cloth, area of coarser cloth Grave goods: 2 jet armlets	RCHME 1962: 83 (ix) Ramm 1971: 193 (28)	YK15: yellow stained mineral-impregnated textile fragments
York, unspecified	YORYM: 2007.6213 Primary ref: 11937		Plaster burial		YK16: orange/yellow residue on inner surface, textile impressions YK17: dark residue on ?external surface of plaster
Heslington Field Found early 1800s	YORYM: 2007.6211 Primary ref: 11935 Finds: H.323, 1-14	Stone sarcophagus Adult female	Plaster burial Textile impressions Grave goods: 'trinkets near shoulder' Hobnail shoe impressions	Wellbeloved 1842 Ramm 1971: 194 (R46)	YK18: orange + dark fragments adhering to plaster

89 The Mount	YORYM: 2007.6209 Primary ref: 11930		Plaster burial, cast showing body contours Embedded bone fragments		YK19: ?soil adhering to plaster near bone fragments YK20: CONTROL associated ?soil sample
York, unspecified	YORYM: 2007.6210 Primary ref: 11934	Lead-lined coffin	Plaster burial Lead ion substitution resulting in very hard grey surface		YK21: minimal adhering residues
York, unspecified	YORYM: 2010.1196 Primary ref: 15419	Stone sarcophagus Heavily tooled Lead liner Late 2nd-4th century AD	Plaster burial Modern contamination i.e. rubbish and plant debris		YK22: debris from base of lead liner YK23: residues from plaster
Catterick	YORYM: 1980.51		Plaster burial, cast showing body contours		YK24: CONTROL, sample from external surface, no residues on inner surface
Railway excavations Found 1877	YORYM: 2010.1219	Stone sarcophagus	Plaster burial Modern modification i.e. bricks to assist display, covered by glass lid Left hand still in situ	RCHME 1962: 82 (iii) Ramm 1971: 193 (15)	YK25: debris from foot end of stone sarcophagus YK26: debris from middle of stone sarcophagus near hand bones YK27: debris from head end of stone sarcophagus
Bishopsgate Street/ Price's Lane Clementhorpe Found 1851	YORYM: 2007.6126 Primary ref: 11149	Previously described as adult female + infant Currently considered to show x3 individuals	Plaster burial Textiles: fine, medium + coarse tabby, 'red'-ribbed strips No grave goods ?removed Lead ion substitution resulting in very hard grey surface	RCHME 1962: 108 (i) Ramm 1971: 194 (R43)	YK28: minimal adhering residues
Railway Station Found 1892	YORYM: 2010.1199 Primary ref: 15421	Lead liner + lid Child c. 7 years	Plaster burial Textile fragments - linen	RCHME 1962: 67(i) Ramm 1971: (1)	No visible residues - not sampled
Trentholme Drive Found 1951-58	YORYM: 1971.303 Grave 196	Stone sarcophagus Sub-adult male c. 14 years Supine, extended	Plaster burial	Wenham 1968: 40 Ramm 1971: 194 (R41)	No visible residues - not sampled

7.10.2 Sample selection

Plaster body-casings and extant sarcophagi from eighteen Roman period inhumations around York were assessed (**Table 7.26**). Twenty-eight samples were collected from sixteen of these burials with the kind permission of the York Museum Trust (YMT), facilitated by Adam Parker. Sample selection and mass were constrained by the materials available which consisted of residues adhering to the inner surface of plaster from thirteen inhumations and grave deposits from the base of two sarcophagi. In the absence of environmental materials, three samples of soils associated with YORKM: 2007.6205i from Mill Mount (YK10), YORYM: 2007.6709 from 89 The Mount (YK19) and YORYM: 1980.51 from Catterick (YK24) were selected as controls. Images, tabulated results and TICs for each sample can be found in **Appendix 6.9** and on **Disc 1, File 6.9**.

7.10.3 Results

7.10.3.1 Control samples

Two of the control samples (YK20; YK24) contained only traces of *n*-alkanes and a solitary *n*-alkanol (C_{26} in YK20). The third, YK10, an apparent mass of soil associated with the plaster burial from Mill Mount (YORYM: 2007.6205i) included a wider range of low abundance lipid species comprising *n*-alkanes, hexadecanoic acid ($C_{16:0}$), a homologous series of HMM *n*-alkanols (C_{24-30}) and the phytosterol, β-sitosterol. This sample is, therefore, consistent with degraded plant matter.

7.10.3.2 Contaminants and ubiquitous lipid species

All of the archaeological samples were minimally soluble in the organic solvent and contaminated by phthalate plasticisers, probably derived from the wrappings in which they had been stored. Many contained little or no organic matter (#9) with the lipid species identified comprising end-products of widespread occurrence (e.g. *n*-alkanes, *n*-alkanols) and unidentified peaks which may be other contaminants or the result of interactions with the plaster matrix. In addition, the samples collected from the sarcophagi currently curated at the rural site near Ricall, just outside York which comprised YORYM: 2010.1196 (YK22-23), YORYM: 2010.1201 (YK12-14) and

YORYM: 2013.1219 (YK25-27) showed evidence of pesticides. This was due to the presence of dieldrin, endrin and what appeared to be benzene hexachloride (banned since 2004). These traces may derive from airborne contamination as a result of local crop spraying or from past treatments undertaken within the museum stores as part of a pest control regime.

The remainder of the samples (#16) contained an assortment of low abundance compounds whose range and patterning suggests a variety of contributors. For example, the *n*-alkanols present probably derive from vegetative matter with the wax esters observed in two samples (YK8; YK14) indicative of an input from the epicuticular leaf waxes of higher plants (Rieley *et al.* 1991). The limited, ubiquitous, SFAs ($C_{16:0;}$ $C_{18:0}$) identified could denote animal and/or plant sources with the octadecenoic acid ($C_{18:1}$) isomers in YK26 more commonly associated with animal fats (Evershed 2008a). The steroidal components support these observations as they represent inputs from both plant (campesterol, stigmasterol, β-sitosterol) and mammalian (cholesterol) tissues. These compounds, in combination, are typical of soil organic matter (Bull *et al.* 2000; Otto *et al.* 2005) and so cannot be considered of archaeological relevance. This is supported by the data from the controls, in particular YK10, the soil from Mill Mount (YORYM: 2007.6205i) although, as the latter did not contain mammalian traces, some contribution from the degradation of intrinsic body tissues or modern handling without gloves is possible in the above mentioned samples.

7.10.3.3 Biomarkers of bitumen

Of greater interest are the biomarkers for bitumen in both samples (YK22; YK23) from YORYM: 2010.1196. These compounds comprise an homologous series of *n*-alkanes ($C_{17-39,}$ C_{31} max), the isoprenoids pristane (Pr) and phytane (Ph) (**Figure 7.64**), traces of regular steranes ($C_{27-31,}$ ααβ + αββ), tri- and tetracyclicpolyprenanes (23/3, 24/3, 24/4) and pentacyclic hopanes ($C_{27-33,}$ isomers) (**Figure 7.65**). Polyaromatic hydrocarbons (e.g. anthracene (AN), phenanthrene (PH), fluoranthene (FL), pyrene (PY), benzoanthracene (BaA), chrysene (CHR)) were also observed.

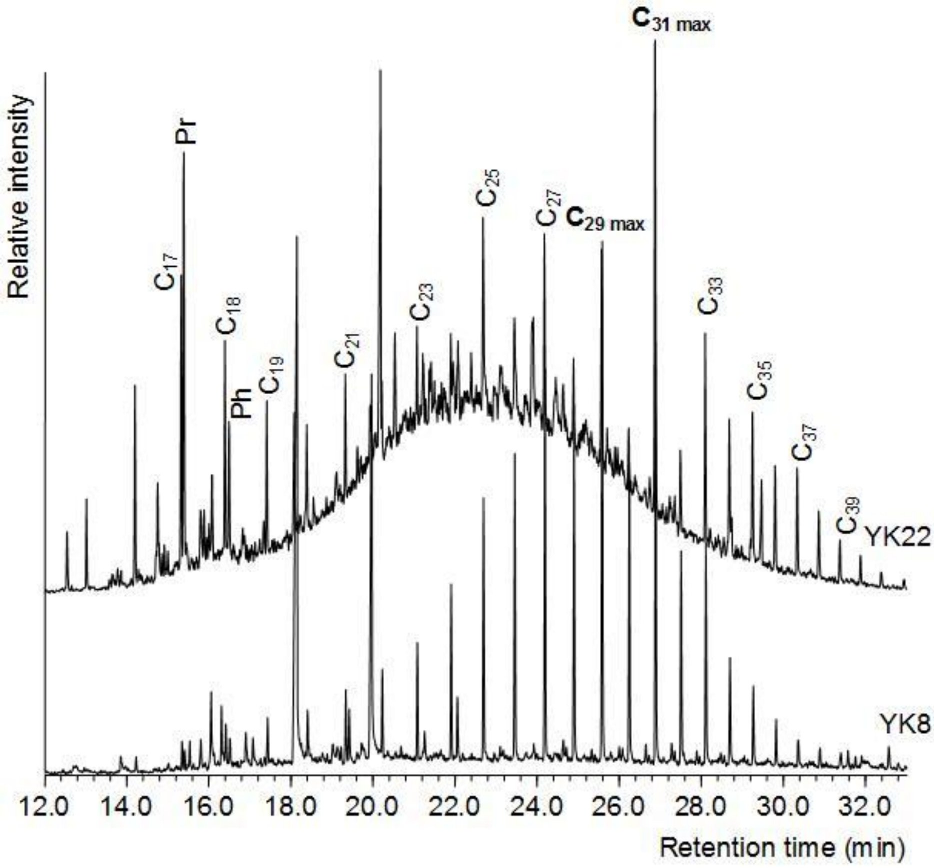


Figure 7.64. Partial XIC (m/z 85) homologous series of *n*-alkanes characteristic of bitumen, plaster burials, YORYM: 2007.6214 (YK8) and YORYM: 2010.1196 (YK22), York, UK.

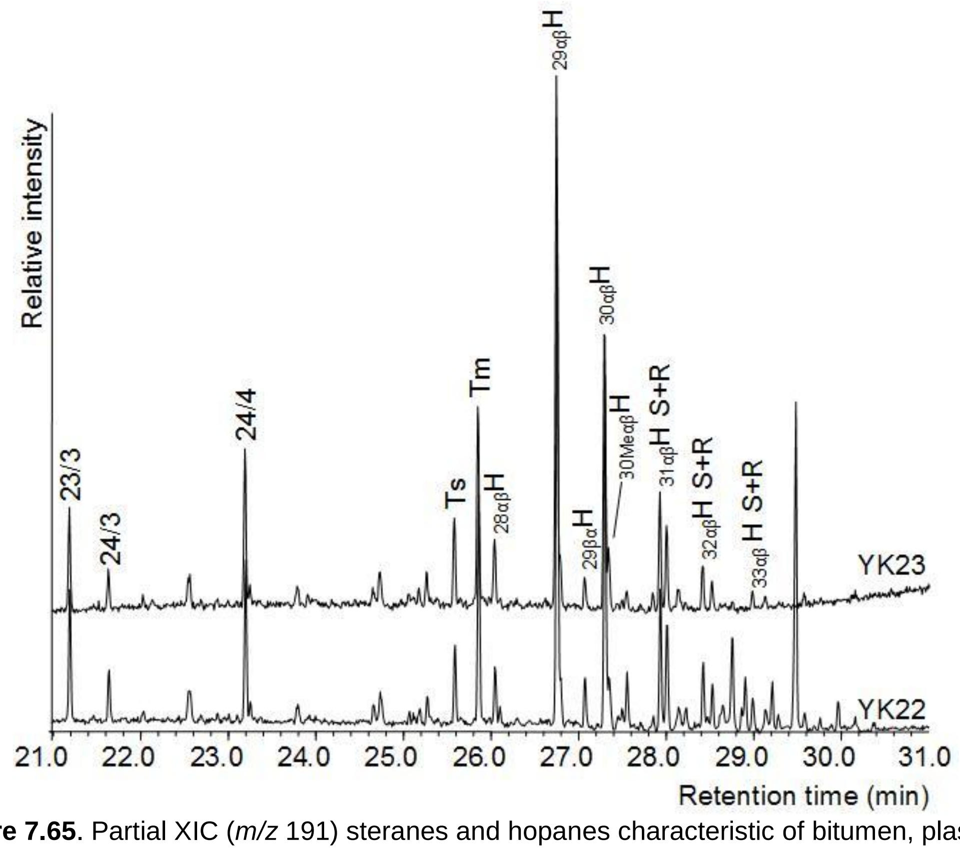


Figure 7.65. Partial XIC (*m/z* 191) steranes and hopanes characteristic of bitumen, plaster burial, YORYM: 2010.1196, York, UK. Peak identifiers relate to **Table 7.27**

Table 7.27. Steranes and hopanes characteristic of bitumen, plaster burial, YORYM: 2010.1196, York, UK. Peak identifiers relate to **Figure 7.65**.

Peak	Compound
23/3	C_{23}-tricyclopolyprenane
24/3	C_{24}-tricyclopolyprenane
24/4	C_{24}-tetracyclopolyprenane
Ts	22,29,30-trisnor-18α-neohopane
Tm	22,29,30-trisnor-17α-hopane
28αβH	17α,21β-29,30-disnorhopane
29αβ/βαH	17α/β,21β/α-30-norhopanes
30MeαβH	2α-methyl-17α,21β-norhopane
30αβH	17α,21β-hopane
31αβH	17α,21β-29,30 homohopane S + R
32αβH	17α,21β-29,30 homodishopane S + R
33αβH	17α,21β-29,30 trishomohopane S + R

Bitumen is the term used to describe the organic solvent-soluble fraction of natural hydrocarbon deposits (Killops and Killops 2005: 128). The organic matter incorporated into bitumen derives from a range of sources, algal, microbial and higher plant, with what survives modified by the depositional environment. Considerable research has been carried out regarding the presence/absence or comparative abundance of various components as indicators of maturation stage, source of contributions (marine, lacustrine or terrestrial) and nature of the source rock (Connan 2012: 90-134; Hunt 1996: 380-393; Maurer *et al.* 2002). In the York samples, the range and relative abundance of the hopanes (C_{27-33} with the αβ isomers prevalent) denotes a bacterial input while the predominance of C_{29} steranes and HMM *n*-alkanes suggests a higher plant contribution. Likewise, a high Pr/Ph ratio (2.7) suggests a mixture of terrestrial and algal source materials (Hunt 1996: 82-95, 407-408). The 'hump' produced by the *n*-alkanes together with a Pr/C_{17} ratio of 1.7 and Ph/C_{18} ratio of 0.7 implies that this is a relatively immature oil (in geological terms). Isomerisation of the steranes (ααβ and αββ forms) and the stereochemistry of the hopanes (αβ, βα and 22S, 22R conformations) suggest some degree of maturity although this could be due to subsequent biodegradation (Hunt 1996: 380-386). Thus, the patterns observed most closely resemble an oil largely derived from terrestrial sources which were, possibly, deposited in aquatic suboxic conditions (Hunt 1996: 542-562).

A similar array of *n*-alkanes (**Figure 7.64**) together with phenanthrenes and hopanes in the dark staining on the plaster body-casing from YORYM:2007.6214 (YK8) also indicate the presence of a petroleum product.

This sample again appears to derive from a terrestrial source based on the *n*-alkane odd-over-even predominance and maximum at C_{29} but from more a mature source rock due to the characteristic, 'hedgehog' effect (Hunt 1996: 380). The absence of steranes, traces of hopanes and survival of only the more resistant aromatic components seems to denote considerable biodegradation (Connan *et al.* 1992). Assessment of certain ratios (AN/PH+AN = 0.19, >0.1 pyrogenic; FL/FL+PY = 0.55, >0.5 pyrogenic; BaA/BaA+CHR = 0.35, >0.35 pyrogenic) indicate that that this petroleum product may had undergone combustion (c.f. Dong *et al.* 2012; Yunker *et al.* 2002).

Likewise, low levels of hopanes were identified in a number of other samples (YK12-14, YORYM: 2010.1201; YK16-17, YORYM: 2007.6213 and YK21, YORYM: 2007.6210) although additional biomarkers for bitumen are absent. As with YK22 and YK23 all of these samples contain traces of what appears to be dieldrin (**7.10.3.2**). It is important, therefore, to stress the potential for modern contamination. This could derive from a range of sources including car exhausts, petrol heaters or pesticides which are often dispersed using a petroleum distillate (Bográn *et al.* 2006). Colonisation by hopane-producing bacteria (Greenwood *et al.* 2006) is another possibility as the bimodal distribution of the *n*-alkanes present in some samples indicates bacterial activity. The use of geochemical techniques to isolate any bound fractions and/or isotopic characterisation might be able to elucidate these issues. No approach can, however, address the date of introduction to these burials due to the nature of fossil hydrocarbon materials and long-term curation of these finds.

7.10.3.4 Terpenic compounds: angiosperm inputs

The final class of compounds present in nine samples collected from six burials were found to be pentacyclic triterpenes due to significant fragment ions at *m/z* 218, 203 and 189 characteristic of oleanane and ursane skeletons with a double bond at C-12 (Modugno *et al.* 2006b). In many of the samples only trace amounts of defunctionalised derivatives rather than suites of diagnostic biomarkers were present making lower level taxonomic determination problematic. Thus, in YORYM:2007.6213 (YK16, YK17), 28-norolean-17-en-3-one and unidentified related compounds (fragments ions at *m/z* 218, 203 and 189) were noted. These appear to represent end products of the degradation of oleanane-skeleton resin acids which, in geochemical studies, are used as markers for terrestrial input from angiosperms (Hunt 1996: 95-97; Pastorova *et al.* 1998; ten Haven *et al.* 1992). Such a limited number of compounds cannot be considered of archaeological relevance especially as traces of hopanes were also observed and the same combination of compounds was present on the inner and outer surfaces of the plaster body-casing which suggests they may represent soil ingress or, more likely, contamination.

Likewise, a range of triterpenic compounds were observed in samples from YORYM:2010.1196 (YK22; YK23), YORYM:2010.1201 (YK14) and YORYM: 2007.6214 (YK8) (**Figure 7.66; Table 7.28**). These comprised the pentacyclic alcohols, β- and α-amyrin (**9, 12**), their oxidation products (**7, 11**) and acetates (**13, 14**). Again, however, they cannot be considered to support the deliberate deposition of plant exudates as these are common components of many plant tissues and evidence for terrestrially-derived bitumen was found in two of these burials (YORYM:2010.1196; YORYM: 2007.6214) alongside hopanes and dieldrin in YORYM:2010.1201, which indicates that this sample should be viewed in a similar light.

Table 7.28. Identification of triterpenic compounds (TMS derivatives), samples from five plaster burials, York, UK based on molecular ion ($M^{+\bullet}$), base peak (BP) and key fragment ions.

Peak	M^{+}.	BP	Key fragment ions	Name of compound
1	394	218	379, 257, 229, 203>189, 175, 161, 147, 119	24-norolean-3,12-diene
2	392	218	377, 281, 253, 207<203, 189, 175, 133	? a noroleantriene
3	394	218	379, 281, 203=189, 175, 161, 147, 133, 119	24-norursa-3,12-diene
4	392	218	377, 281, 253, 207> 203, 189, 147, 133	?a norursatriene
5	498	218	393, 327, 279, 257, 203>189/190, 175, 147, 121	3-*epi*-β-amyrin
6	498	218	483, 408, 382, 203<189/190, 175, 161, 147, 121	3-*epi*-α-amyrin
7	408	232	393, 353, 273, 255, 218, 203, 189, 161, 135	24-norursa-3,12-dien-11-one
8	424	218	409, 391, 367, 313, 257, 203, 189, 175, 161, 109	β-amyrenone
9	498	218	483, 468, 408, 393, 203, 189, 175, 161, 129, 119	β-amyrin
10	410	163	395, 279, 257, 218, 203, 191, 175, 133, 119	28-norolean-17-en-3-one
11	424	218	409, 391, 367, 313, 257, 203, 189, 175, 161, 109	α-amyrenone
12	498	218	468, 408, 393, 203<189, 175, 161, 135, 119	α-amyrin
13	468	218	453, 408, 393, 257, 231, 203>189, 175, 119	β-amyrin acetate
14	468	218	453, 408, 393, 249, 231, 203<189, 161, 121	α-amyrin acetate

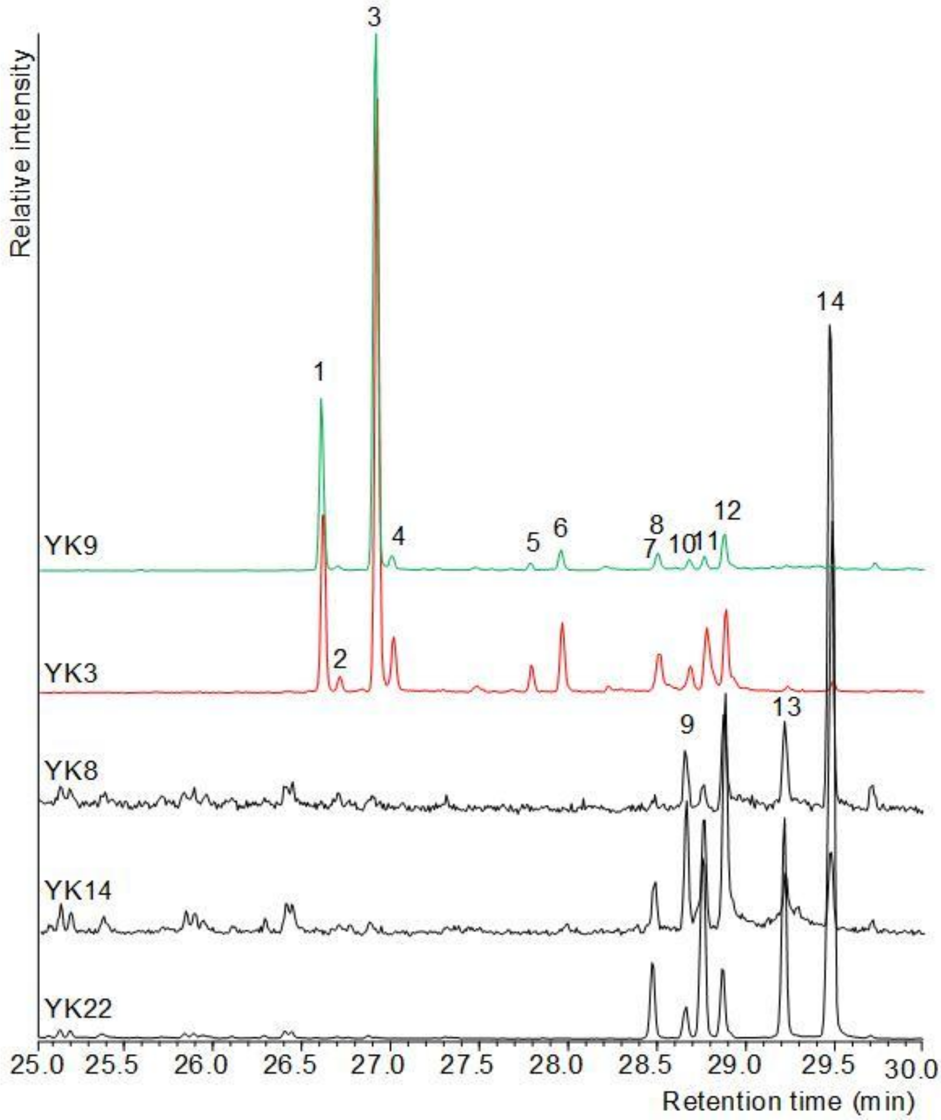


Figure 7.66. Partial XIC (*m/z* 218) triterpenic compounds, in five plaster burials, YORYM:2010.1196 (YK22), 2010.1201 (YK14), 2007.6214 (YK8), 2007.6206 (YK3), 2007.6205i (YK9), York, UK. Peak identifiers relate to **Table 7.28**.

In contrast, the residue from the cranial region of the plaster (YK9) from YORYM:2007.6205i may be of archaeological significance as it contained a broader and more abundant suite of triterpenic compounds (**Figure 7.66; Table 7.28**) which were not present in the corresponding soil control (YK10). These comprised the triterpenic alcohols, β- and α-amyrin, their epimers and a number of oxidation products with ursane (α) skeleton isomers more abundant than oleanane (β). The remainder of the triterpenes consisted of olean-12-ene and urs-12-ene derivatives with a methyl group at C-17 as distinguished by a base peak at *m/z* 218 (c.f. Budzikiewicz *et al.* 1963). Unfortunately, no precursor resin acids were present to enable the source to be definitively identified. Comparison with the published literature, however, indicates that this sequence reflects the range of compounds observed in degraded gum-resins from the genus *Boswellia*, with the 24-nordienes considered diagnostic derivatives of the boswellic acids (Baeten *et al.* 2014; Modugno *et al.* 2006b; ten Haven *et al.* 1992).

An identical series of triterpenic compounds, potentially indicative of frankincense, were also present in two of the residue samples (YK3; YK5) from YORYM:2007.6206 (**Figure 7.66**). In addition, sesquiterpenes were noted in YK4, the third sample from this burial (**Figure 7.67**). These LMM moieties are an unexpected find as they are highly volatile and so rarely survive over archaeological time. Due to their low abundance and similar fragmentation patterns definitive identification is problematic and based, in part, on elution order. Those most clearly characterised (e.g. curcumene, cuparene, longipinene and related compounds) are of widespread occurrence but are inconsistent with frankincense.

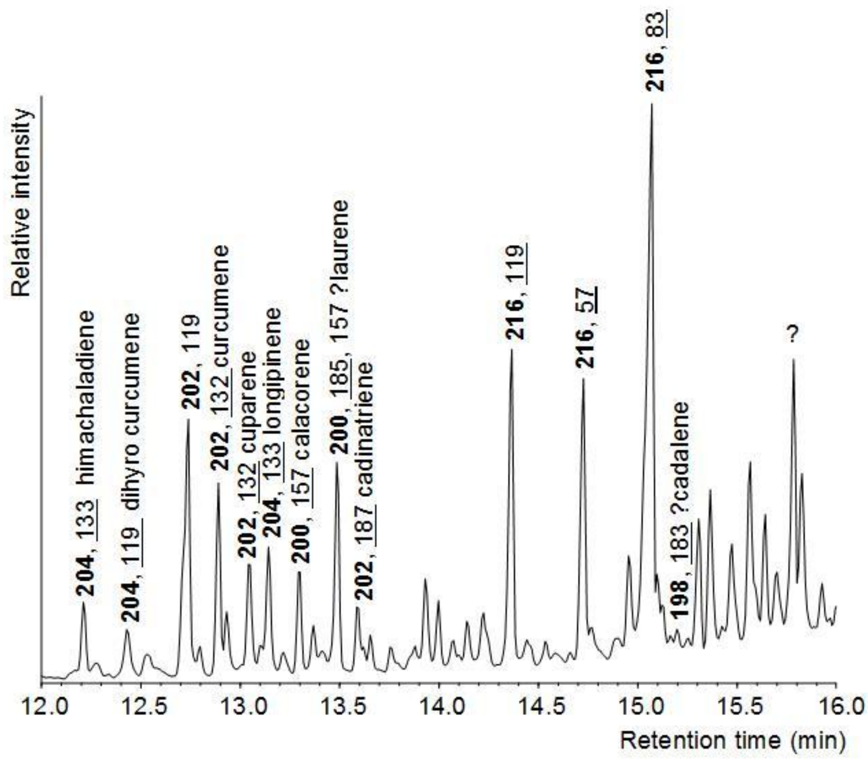


Figure 7.67. Partial TIC (12-16 min) sesquiterpenes and related compounds, residue (YK4), plaster burial YORKM:2007.6206, York, UK. Molecular ion in bold, base peak underlined.

They have, however, been reported in the resinous wood and exudates of members of the Cupressaceae (Enzell and Erdtman 1958; Kamatou *et al.*

2010) and Pinaceae (Colombini *et al.* 2000; Koller *et al.* 2003). Tentative classification of other moieties as benzocycloheptenes ($M^{+\cdot}$ 216 and 218) as described by Buckley *et al.* (2004) appear to support the presence of a conifer product (Langenheim 2003: 329-331). Although a mixture of plant exudates could have been incorporated in this burial it seems more likely that these sesquiterpenes derive from the wooden coffin in which this individual was interred. This is supported by the position of the residue near a crack penetrating from the outer surface of the plaster and an accompanying note stating that fragments of the coffin wood had been identified as cedar.

As regards the presence of frankincense, comparison was made with modern botanically and geographically identified *Boswellia* spp. resins (**Appendix 3.3**). Evaluation of the triterpenic region demonstrated that the neutral derivatives in the archaeological samples were identical to those in the modern reference materials (with the exception of 28-norolean-17-en-3-one, present in the former) with the α-isomer consistently more prominent than the corresponding β-isomer. Although contamination during their subsequent curatorial history cannot be excluded, the application of a substance that could mimic frankincense to both plaster body-casings, the first from a stone cist found in 1848 south-east of the current railway station and the second, from an inhumation on Mill Mount, seems unlikely.

The final evidence for pentacyclic triterpenic compounds comes from three samples (YK25-27) obtained from the base of the sarcophagus containing burial YORYM:2010.1219 which had the plaster body-casing and left hand bones still in situ (**Figure 7.68**). Key fragment ions at *m/z* 189, 218, 279 and 320 with a base peak at *m/z* 203 are characteristic of olean-12-enes (Assimopoulou and Papageorgiou 2005a) with the dominant compound found to be oleanonic aldehyde (olean-12-en-3,28-dienone, produced by the reduction of oleanonic acid) (**Figure 7.69; Table 7.29**). This was preceded by a trace of oleanonic acid in YK25 and YK26 (**Figure 7.70**). In addition, 28-norolean-12-en-3-one (**1**), formed by the decarboxylation of oleanonic acid, together with the more stable 28-norolean-17-en-3-one (**4**), the result of a subsequent hydrogen shift (Pastorova *et al.* 1998) were present alongside friedoolean-3-one (**7**), a defunctionalised oleanane triterpenoid with a migrated methyl group (Shan *et al.* 2013).

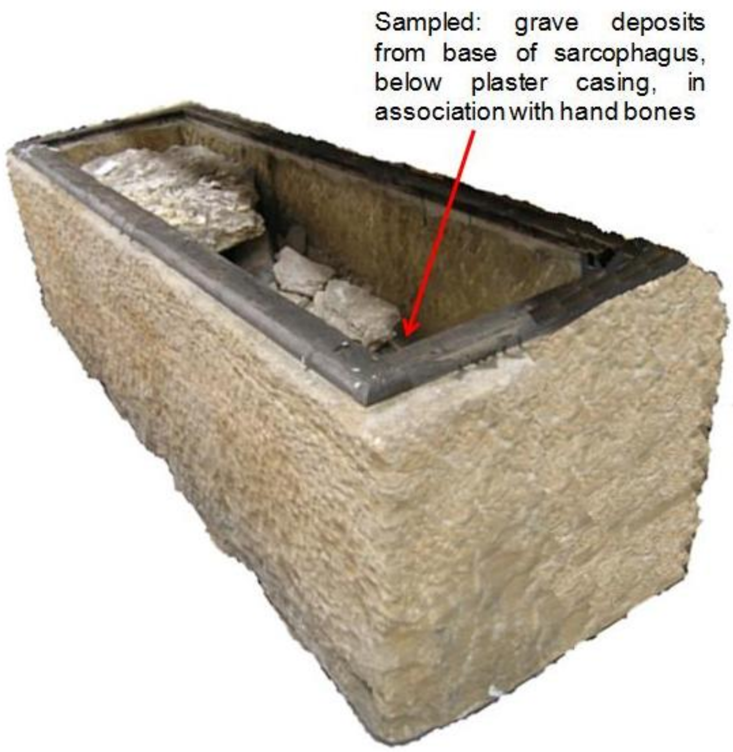


Figure 7.68. Stone sarcophagus containing plaster burial, YORYM: 2010.1219, York, grave deposits from base still associated with in situ left hand bones sampled (Author).

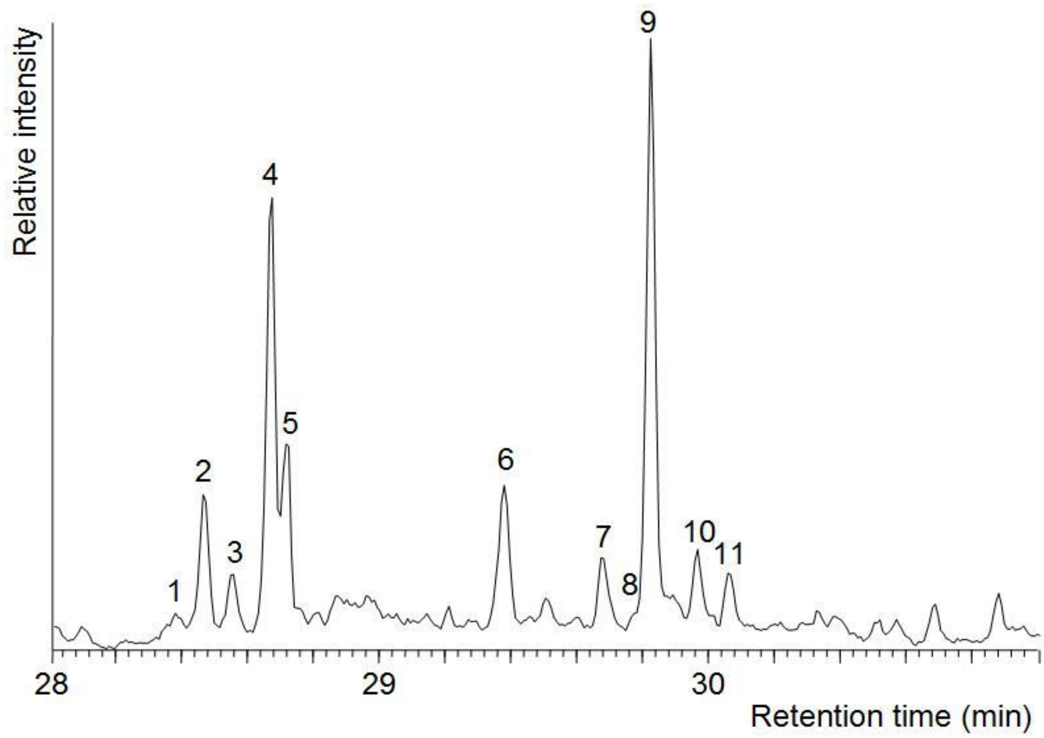


Figure 7.69. Partial XIC (*m/z* 203) triterpenic compounds, grave deposit (YK26), plaster burial YORYM.2010.1219, York, UK. Peak identifiers relate to **Table 7.29**.

Table 7.29. Identification of triterpenic compounds (TMS derivatives), plaster burial YORYM.2010.1219, York, UK based on molecular ion ($M^{+\bullet}$), base peak (BP) and key fragment ions.

Peak	$M^{+\cdot}$	BP	Key fragment ions	Name of compound
1	410	204	395, 239, 218, 189, 175, 133, 121	28-norolean-12-en-3-one
2	424	218	409/6, 391, 239, 203>189, 119, 95	β-amyrenone
3	408	408	393, 357, 215, 202, 189, 163, 105	?nordienone
4	410	163	395, 279, 257, 218, 203, 133, 119	28-norolean-17-en-3-one
5	408	408	393, 258, 229, 203, 189, 173, 119	28-norolean-12,17-dien-3-one
6	422	422	407, 216, 203, 189, 175, 145, 131	olean-12,17-dien-3-one
7	426 ?	69 143	411, 393, 273, 205, 163, 123, 109	friedoolean-3-one dammarane co-eluting
8	526	203	511, 302, 273, 218, 205, 191, 163	oleanonic acid
9	438	203	409, 232, 219, 189, 175, 145, 105	oleanonic aldehyde
10	?438	203	409, 391, 232, 218, 189, 143, 161	?oleanolic aldehyde
11	?512	257	497, 422, 407, 231, 203, 189, 119	observed in modern *Pistacia* spp.

Although no definitive biomarkers such as moronic acid were identified, this combination of compounds closely reflects those observed in aged *Pistacia* spp. resins (Modugno *et al.* 2006b). The reduction in the resin acids does not appear to be the result of heating due to the relatively low abundance of 28-norolean-17-en-3-one and presence of ocotillones (oxidised dammaranes with a base peak of *m/z* 143) (c.f. Serpico and White 2000; Stern *et al.* 2003; van der Doelen *et al.* 1998). Similar age-related changes have been observed in curated modern *Pistacia* spp. exudates and comparison of the archaeological data with such reference materials showed a clear correspondence in the compounds present (**Appendix 3.2**).

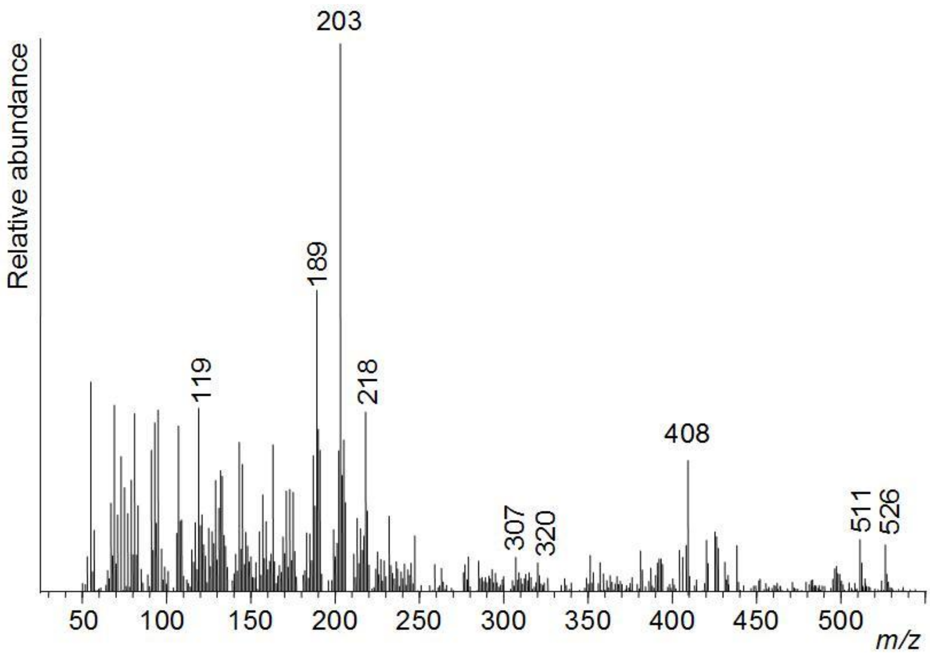


Figure 7.70. Mass spectrum (29.8 min) assigned as oleanonic acid, grave deposit (YK26), plaster burial YORYM:2010.1219, York, UK.

7.10.4. Summary and interpretation of findings

Many of the samples contained little or no organic matter while the remainder suffered with issues of contamination. A key consideration in archaeological residue analysis, this was of particular concern here as the majority of these plaster burials had been found in the 1800s and so had undergone an extended curational history which in some cases involved open-air display. The control samples, although not ideal, provided some indications with regards to background lipid content. This comprised low abundance degradation products (*n*-alkanes, *n*-alkanols, SFAs) of widespread occurrence which reflected input sources (microbial, plant and animal) commonly observed in soil organic matter. Thus, where these moieties were present in the archaeological samples they were discounted.

A range of contaminants were also observed with phthalate plasticisers present in all samples. These are a ubiquitous modern problem and easily identified. Traces of pesticides (e.g. dieldrin) are more unusual and may derive from crop spraying or pest management. This fits well with the current rural storage location of these sarcophagi and the need to maintain the integrity of the museum collection in the face of insect infestations. The source of the hopanoids is more complex to unravel. Traces of hopanes together with a bimodal or limited series *n*-alkanes may simply reflect the actions of hopane-producing bacteria (e.g. YORYM:2007.6213). Nonetheless, the presence of bitumen derived from terrestrial input sources was confirmed in two burials (YORYM:2007.6214; YORYM:2010.1196) due to the range of hopanes in combination with an extensive homologous series of *n*-alkanes and other markers such as steranes and polyaromatic hydrocarbons. When this substance came into contact with the plaster cannot be ascertained, however, given the widespread use of petroleum products in the modern world and context of the samples. These findings are not, therefore, considered of archaeological relevance.

A similar situation exists with regards to the triterpenic compounds. Where only a limited number of neutral end-products (e.g. 28-norolean-17-en-3-one) or commonly-occurring moieties (e.g. β- and α-amyrin) are observed they must simply be viewed as representative of input from flowering plants. Such compounds are not sufficiently diagnostic of source and so could derive from modern soil ingress, terrestrially-sourced bitumen (of modern or ancient introduction) or floral offerings rather than the use of resins. Suites of triterpenes are a different matter. These can be related to source materials with far greater confidence although diagnostic resin acids are still required for definitive determinations. Thus, the results from YORYM:2007.6205i, 2007.6206 and 2010.1219 provide strong indications rather than conclusive evidence for the presence of two exotic triterpenoid-containing resinous exudates, *Pistacia* spp. resins and *Boswellia* spp. gum-resins.

To summarise, analysis of extant samples from sixteen 4th century AD plaster burials discovered around York, North Yorkshire, UK revealed suites of triterpenoids in three inhumation burials with traces of degradation products in a further three. These findings suggest that exudates of archaeological origin comprising *Pistacia* spp. (mastic/terebinth) resins and frankincense (*Boswellia* spp.) were incorporated within a number of these burials. Chemical evidence of bitumen was also recovered although the date of its inclusion cannot be determined. This confirms York as the most northerly example of the use of resins as part of late Roman mortuary practices.

7.11 Case Study 10: Mersea Island barrow, Essex (Brettell *et al.* 2015b)

7.11.1 Background

Mersea Island, situated off the coast of Essex, is one of a small number of tidal islands found around the UK. Accessible from the mainland via a causeway called the Strood (which floods at high tide), the island is positioned beside the Purfleet Channel at the confluence of the estuaries of the Blackwater and the Colne (Tyler 2009). Evidence of human activity dates from prehistory with finds of worked flint, ceramics and PRIA salt extraction sites known as the Red Hills, with the latter often accompanied by piles of oyster shells (Hazzledine Warren 1913; Karbacz 2012: 12-13). Settled in the Roman period, if not before, the remains of a number of brick and stone-built buildings and tessellated pavings, possibly a villa site, have been identified near the church of SS. Peter and Paul (Karbacz 2012: 15-16). A cremation burial of a child in a globular green glass vessel with lead seal over the mouth was also recovered in the vicinity, not far from the foundations of a round tower or wheel tomb. Enclosed by flue-tiles, this urn and accompanying ceramic oil lamp, probably manufactured in northern Italy in the 2nd century AD, was located below a brick-built structure (Benton 1924).

The most significant ancient landmark, however, comprises the extant remains of an earth-built tumulus located on high ground and known as the Mersea Island barrow or Mersea Mount (TM 060 150; Karbacz 2012: 14). One of a number of round barrows in the region, excavation of others such as those at Bartlow Hills on the Essex-Cambridgeshire border had previously revealed that these structures often contained elaborate cremation burials dated to the early Roman period (Gage 1834, 1836). Thus, the mound at Mersea, which had inspired all sorts of local folk tales and featured in the gothic novel, *Mehalah*, penned in 1888 by the Reverend S. Baring-Gould, became the focus of antiquarian interest in the early 1900s (Howlett 2013: 9-11). Excavations were carried out in 1912 by Samuel Hazzledine Warren, a geologist working under the auspices of the Morant Club (Hazzledine Warren 1913). A trench was dug into the mound and a central shaft opened up to locate the burial chamber. This consisted of an outer structure founded on a single layer of boulders covered by a dome formed from courses of roofing tiles **(Figure 7.71a)**.

The chamber itself, which extended below ground level, had foundations constructed of two rows of boulders and some tiles set in mortar. These supported walls built of horizontally laid roofing tiles. Two more flanged tiles acted as a floor to the main area which contained a square lead casket with two wooden boards, still in good condition, laid over the top to form a lid **(Figure 7.71b)**. This ossuary held an intact globular green glass vessel which had been employed as a cremation urn and was part-filled with a liquid (Hazzledine Warren 1913; **Figure 7.71c**). The lead casket and its contents were lifted in their entirety and taken to Colchester Castle Museum where they were studied by the curator, Arthur Wright (Howlett 2013: 15). Finds from the mound included fragments of various ceramics including imported wares alongside what appeared to be locally-made coarser pottery, crushed oyster shells and briquetage, broken pieces of vessels used in the processing of salt water. Dated to the late 1st-2nd centuries AD, these, alongside some earlier flint flakes, were interpreted as having been redeposited during construction of the mound (Hazzledine Warren 1913). The glass vessel, itself, was considered by Wright to date to the late 1st century AD and initial evaluation of the cremated remains indicated that these belonged to an adult. They were, however, on display in Colchester for a hundred years erroneously labelled as those of a child presumably due to confusion with the 'wheel-tomb' find (Howlett 2013: 15).

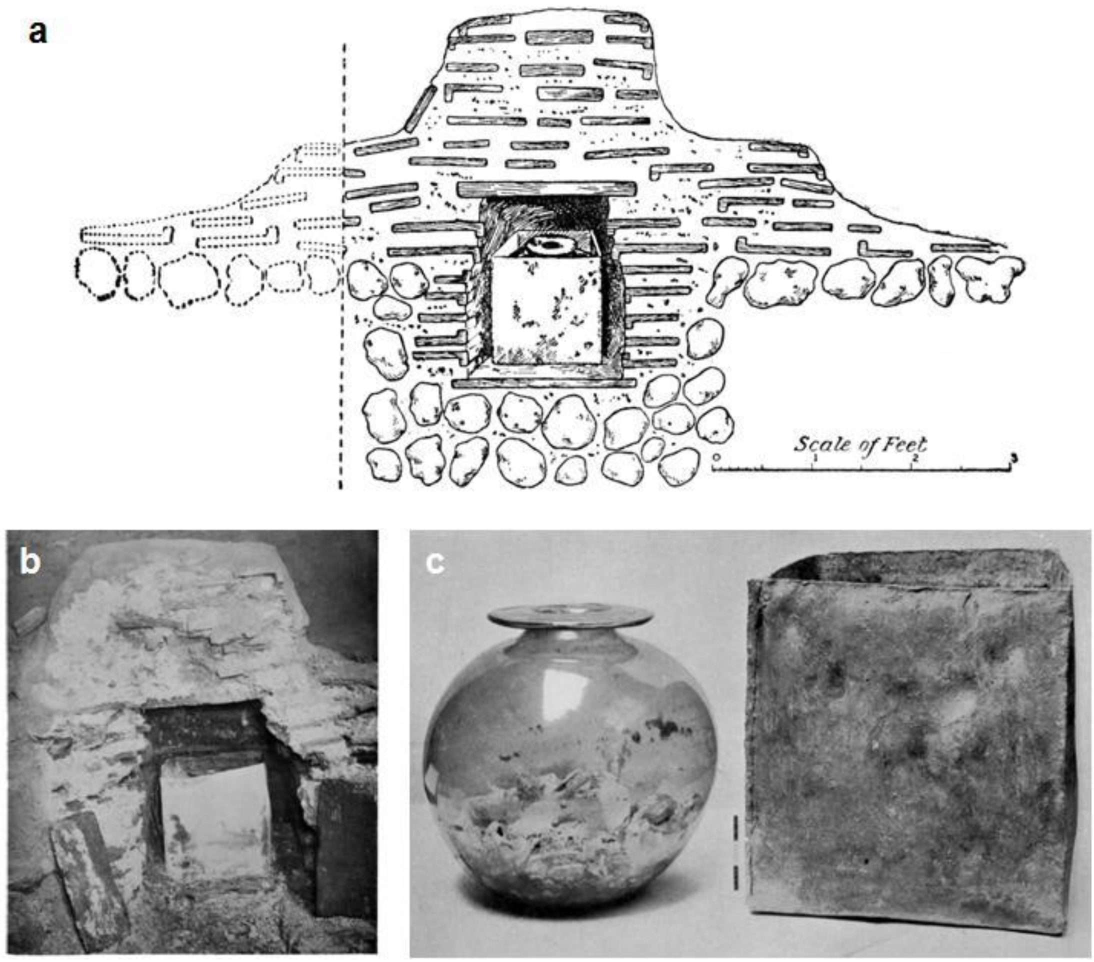


Figure 7.71. Burial chamber and contents, Mersea Island barrow, Essex, UK: a. scale drawing of the tomb structure, Hazzledine Warren 1913: 129, Figure 2; b. lead ossuary in situ within the tile structure with the wooden covering boards removed, Hazzledine Warren 1913: 128, Plate D; c. glass vessel containing the cremated remains and the lead ossuary, Hazzledine Warren 1913: Plate E, 131.

In 2012, the Mersea Mount cremation vessel and its contents were returned to the island for a centenary exhibition at the museum. This raised awareness of the significance of the barrow. Re-evaluation of the finds, in particular the presence of chevron-decorated grey-ware sherds (possibly from a CAM 278 form vessel), suggested that the barrow may have been erected at a somewhat later date (c. 140-190 AD) (Benfield and Black 2014). In addition, a campaign for re-analysis of the human remains led to generous donations from visitors and locals and a grant from the Association for Roman Archaeology which enabled J. McKinley, Senior Osteoarchaeologist at Wessex Archaeology, to be employed (Howlett 2013: 70). This research revealed a substantial quantity (1730.50 g) of variably oxidised human bone which represented a considerable proportion of the remains of a robust adult, probably a male, aged around 35-45 years old. Skeletal markers indicative of regular strenuous activity or related to the evidence for diffuse idiopathic skeletal hyperostosis (DISH) on the cervical and thoracic vertebrae and age-related degenerative changes were noted. Examination also revealed amorphous masses of yellow-white material (92.9 g) with more coating the surfaces of the bone. This gave off a strong aroma and was separated from the cremated remains by wet-sieving (McKinley 2014; **Figure 7.72a**).

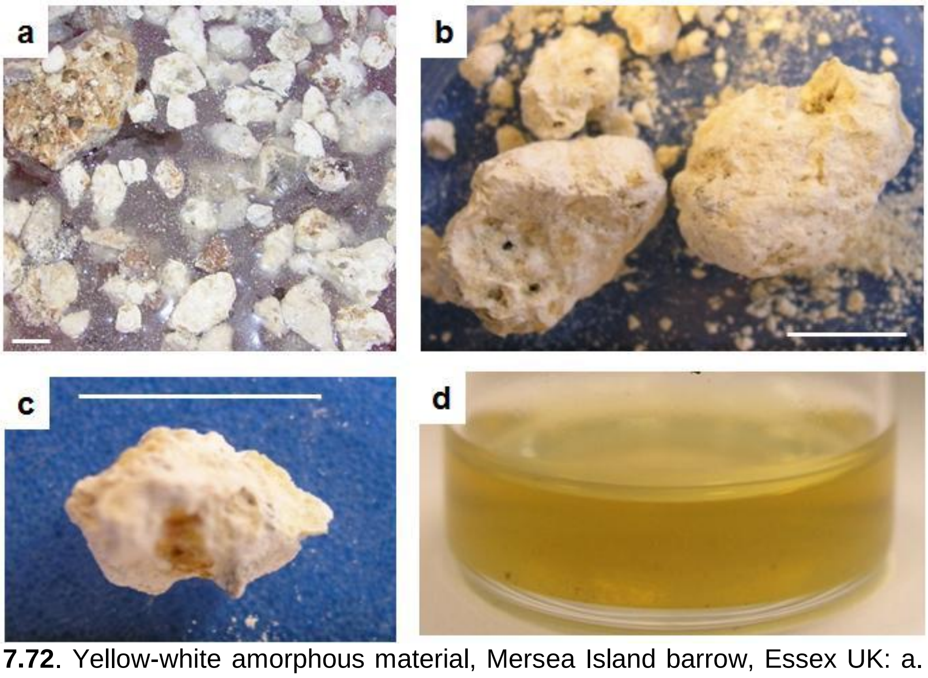


Figure 7.72. Yellow-white amorphous material, Mersea Island barrow, Essex UK: a. during wet-sieving; b. portion received for organic residue analysis; c. sub-sample split for analysis showing inner orange; yellow coloured total lipid extract obtained (Author). Scale bars: 1 cm.

7.11.2 Sample selection

A quantity of this substance was submitted for chemical analysis (**Figure 7.72b**). Ensuring that a portion was retained for future assessment, three sub-samples (MS1-3) were broken off from the larger masses supplied. Initial evaluation was undertaken using attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) followed by full characterisation by GC-MS. Additional images and details can be found in **Appendix 6.10**, TICs of the silylated extracts on **Disc 1, File 6.10** and comparative data from modern reference resins in **Appendix 3.3.**

7.11.3 Results

7.11.3.1 Sample description

Visual assessment of the portions separated from the larger masses revealed orange fragments embedded within a yellow/white amorphous outer matrix (**Figure 7.72c**). These sub-samples dissolved fully in the organic solvent to produce a yellow liquid with a powerful scent (**Figure 7.72d**).

7.11.3.2 Initial findings: ATR-FTIR

Initial analysis using ATR-FTIR spectroscopy provided near identical spectra from both the white and orange materials (**Figure 7.73**). These demonstrated bands characteristic of polycyclic hydrocarbons with oxygen-containing functional groups which comprised:

- a shifted broad stretch (ν) due to O-H groups ~3500-3300 cm^{-1};
- strong ν(CH_2) and ν(CH_3) bands ~2930-2870 cm^{-1};
- strong ν(C=O) bands ~1750-1700 cm^{-1} in triterpenoid and ~1700-1690 cm^{-1} in diterpenoid resins due to carboxylic acid (COOH) groups;
- ν(C=C) shoulder ~1650 cm^{-1} and ring vibrations ~1550-1480 cm^{-1};
- CH_2 and CH_3 group bending (δ) ~1460-1450 and ~1375-1385 cm^{-1};
- ν(C-O) bands between ~1275-1240 cm^{-1} and ~1115-1010 cm^{-1};
- C-H out of plane deformations ~880-890 and ~840-820 cm^{-1};
- CH_2 group rocking (ρ) modes ~730-740 cm^{-1}.

This combination closely resembles the spectra obtained from triterpenoid resins, in particular the presence of a C=O stretching band above 1700 cm^{-1}, although additional sharp bands at 983 cm^{-1} and 658 cm^{-1} suggested a possible admixture of aromatics as such features have been noted in diterpenoid resins (Archier and Vieillescazes 2000; Bruni and Guglielmi 2014; Colombini *et al.* 2009; Derrick *et al.* 1999: 82-108; Shearer 1989).

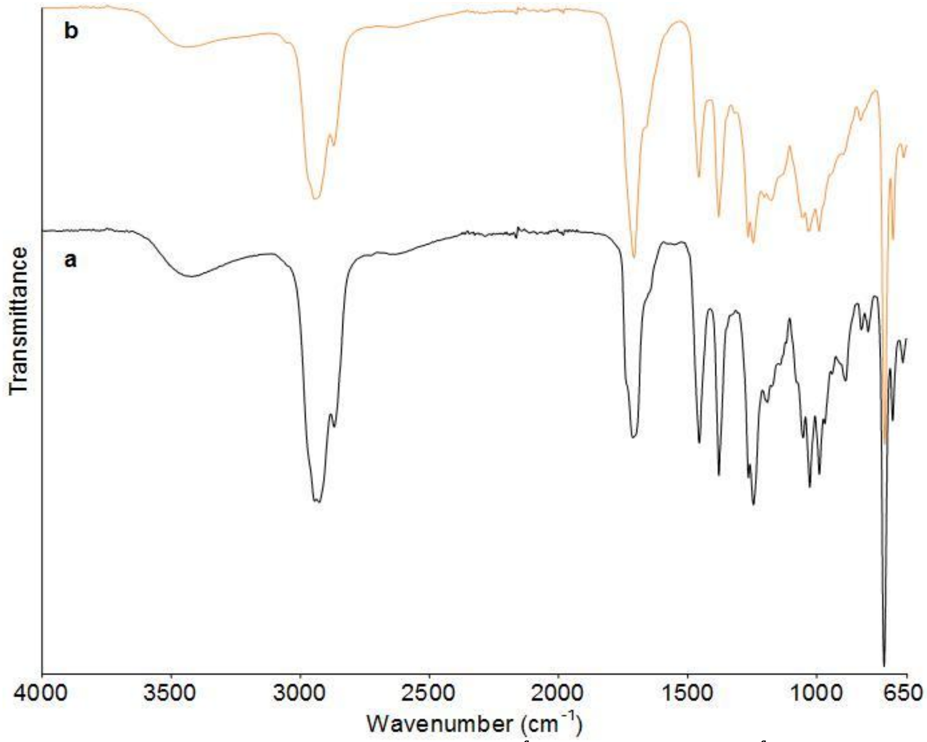


Figure 7.73. ATR-FTIR spectrum, 4000-650 cm^{-1}, 16 scans, 4 cm^{-1} spectral resolution, cremation burial, Mersea Island barrow, Essex, UK: a. outer yellow-white amorphous fraction; b. inner orange fraction.

7.11.3.3 Characterisation: GC-MS

All three sub-samples were found to contain traces of ubiquitous saturated ($C_{16:0}$; $C_{18:0}$) and unsaturated ($C_{18:1}$) carboxylic acids. These are present in most plant and animal tissues, including resinous exudates. In addition, compounds characteristic of natural resins were identified. These consisted of a range of terpenoids from all of the four main groups: mono-, sesqui-, di- and triterpenes (**Figure 7.74**). The presence of LMM mono- and sesquiterpenes was unexpected as these highly volatile components rarely survive in aged resinous materials. Those tentatively identified are listed in **Table 7.30**. Unfortunately, their common occurrence means that they are of limited diagnostic potential. The range present, however, as with the FTIR data, suggests that they could derive from a mixture of angiosperm and conifer resins as sesquiterpenes are often in abundance in the former and monoterpenes in the latter (Langenheim 2003: 36-38). Identification of the HMM compounds which consisted of diterpenoids with abietane and pimarane skeletons alongside cembrene and verticillane-based diterpenes and triterpenic constituents with oleanane and ursane skeletons confirmed this hypothesis. This combination does not occur in nature.

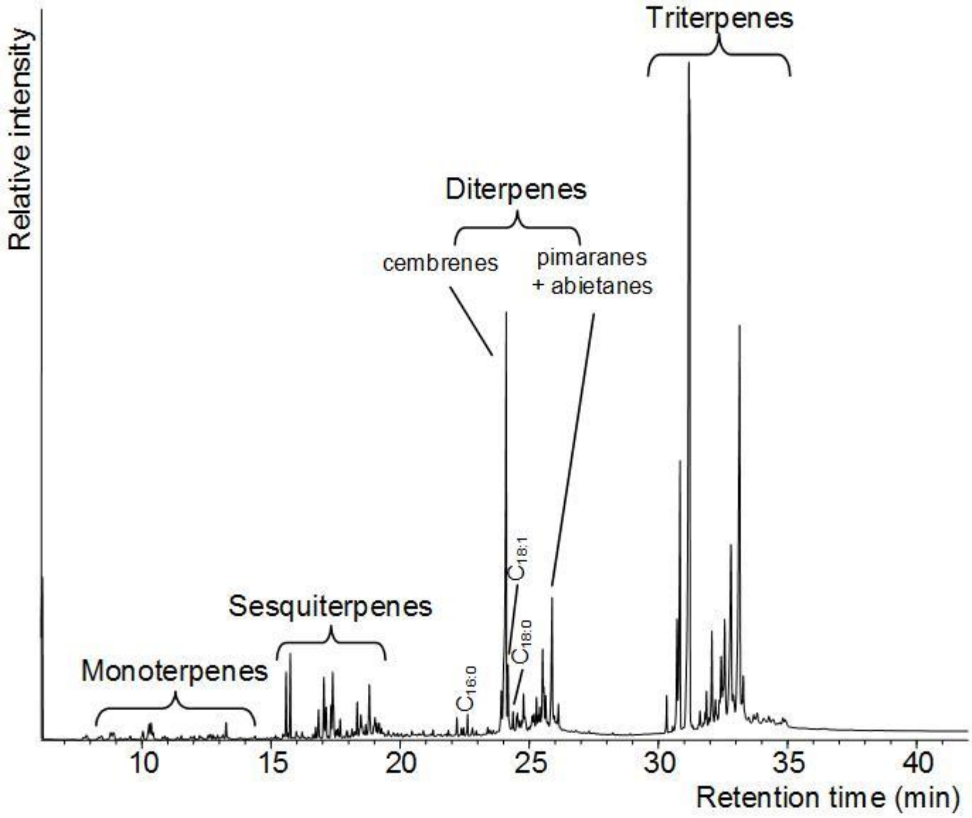


Figure 7.74. TIC range of terpenic compounds present, amorphous residue (MS3), cremation burial, Mersea Island barrow, Essex, UK.

Table 7.30. Mono- and sesquiterpenes, amorphous residue, cremation burial, Mersea Island barrow, Essex, UK.

Monoterpenes	Sesquiterpenes
α-pinene (1R + 1S)	α-copaene
β-pinene	β-elemene
α-phellandrene	β-caryophyllene
camphene	α-caryophyllene
menthene	aromadendrene
o-cymene + β-terpinyl acetate	γ-muurolene
β-phellandrene	eudesma-4(14),11-diene
δ-3-carene	α-muurolene + α-selinene
γ-terpinene	γ-cadinene
cymenene + terpinolene	δ-cadinene
ocimene	calamenene
dihydrocarvone	α-calacorene
pinocarvone	carophyllene oxide
isocineole (1,4-cineole)	?longifolene
α-terpineol	τ-muurolene
verbenone (2-pinen-4-one)	

The diterpenoid resin acids identified (pimaric (PM), sandaracopimaric (SDPM), *iso*pimaric (IPM) and abietic (AB) acid; **Figure 7.75; Table 7.31**) are biomarkers for exudates from members of the Pinaceae, a conifer family which includes pines, firs, spruces, cedars and larches (Colombini and Modugno 2009; Langenheim 2003: 37, 54-59). All of these genera contain a similar range of compounds whose relative abundances vary with environmental factors. In archaeological samples the homogenising effect of degradation pathways must also be considered. Thus, a lower level of taxonomic classification is rarely reliable unless, for example, labdanes characteristic of resins from *Abies* spp. (firs) and *Larix* spp. (larches) are identified (c.f. Mills and White 1999: 101). These compounds were not observed here. Nonetheless, the relative abundance of abietic acid, represented here by the precursor acid itself (AB) and its degradation products, dehydroabietic acid (DHA) and didehydroabietic acid (DDHA), suggests that the source may be a *Pinus* spp. resin.

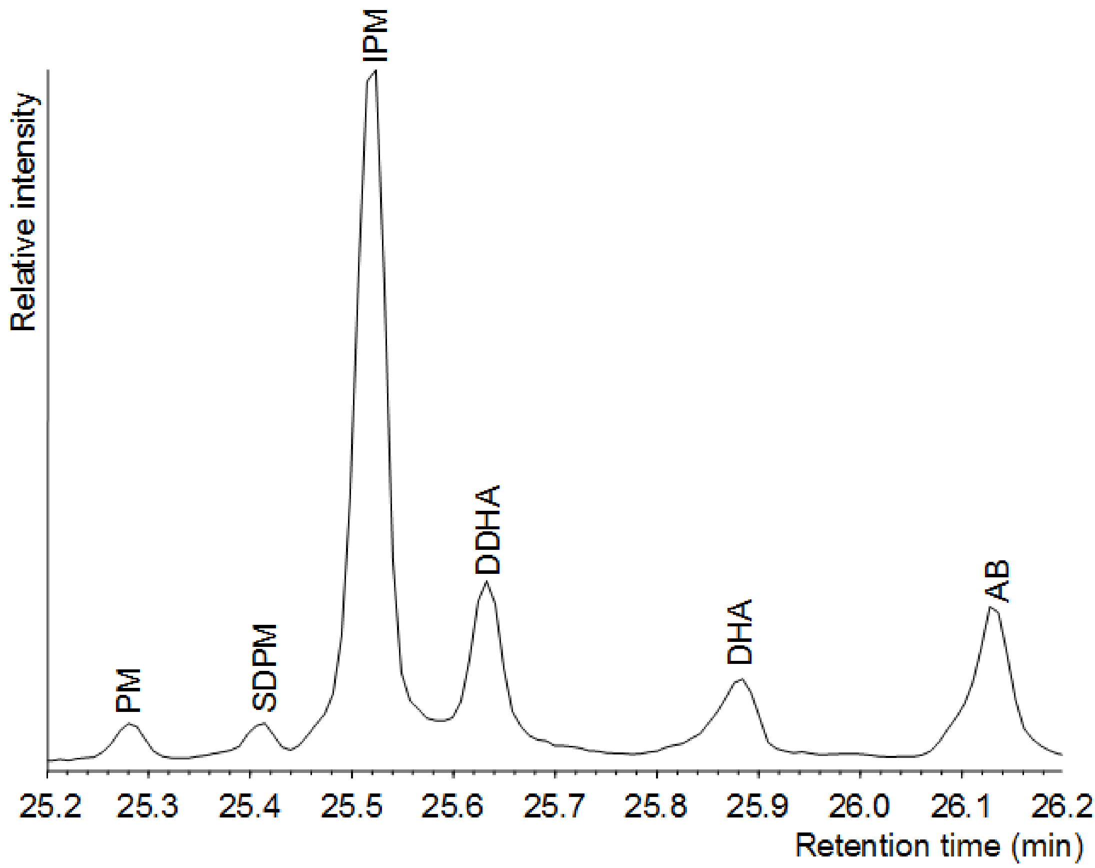


Figure 7.75. Partial XIC (*m/z* 241) diterpenoids, amorphous residue (MS3), cremation burial, Mersea Island barrow, Essex, UK. Peak identifiers relate to **Table 7.31**.

Table 7.31. Identification of diterpenoids (TMS derivatives), amorphous residue, cremation burial, Mersea Island barrow, Essex, UK based on molecular ion ($M^{+\bullet}$), base peak (BP) and key fragment ions.

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
PM	374	73	121, 133, 191, 207, 257, 299, 359	Pimaric acid
SDPM	374	121	73, 91, 143, 241, 257, 359	Sandaracopimaric acid
IPM	374	241	73, 105, 143, 256, 257, 359	Isopimaric acid
DDHA	370	237	73, 103, 143, 195, 209, 252, 355	Didehydroabietic acid
DHA	372	239	73, 129, 143, 171/3, 185, 240, 255	Dehydroabietic acid
AB	374	256	73, 105, 185, 213, 241, 257	Abietic acid

The survival of a range of precursor acids, which are dominant in fresh Pinaceae resins but less frequently observed in archaeological samples, indicates that these deposits were relatively well preserved. Some evidence of alteration was demonstrated by the presence of DHA, DDHA and traces of defunctionalised abietane derivatives. Extensively modified, highly stable, end products of the degradation pathways of the resin acids, such as retene or methyl dehydroabietate, were, however, not observed. This combination suggests that the resin was naturally aged and had not been heated, at least not to the level of a tar or pitch. Comparison with data obtained from the analysis of various modern Pinaceae products (**Appendix 3.1**) confirmed these findings. This conifer resin appeared to be the lesser component in the mixture as the chromatograms were dominated by triterpenic compounds with a strong contribution from cembrene-skeleton diterpenes.

Thus, the major contributor was characterised by pentacyclic triterpenic compounds with olean-12-ene and urs-12-ene skeletons due to significant fragment ions at *m/z* 189, 203, 218 and 292. Such compounds are characteristic of exudates from members of the extensive resin-producing angiosperm family, the Burseraceae (Howes 1949: 86-87; Mathe *et al.* 2009; Tucker 1986). In this instance, however, the abundant neutral degradation products 24-noroleana-3,12-diene (**1**), 24-norursa-3,12-diene (**2**), corresponding nortrienes and 24-norursa-3,12-dien-11-one (**5**) (**Figure 7.76; Table 7.32**) provided a strong indication as to source. These compounds are distinguished by a methyl group at C-17 which results in a base peak at *m/z* 218 (Budzikiewicz *et al.* 1963) and are characteristic of resins from the genus *Boswellia*, better known as frankincense (Başar 2005: 117-119; Mathe *et al.* 2007).

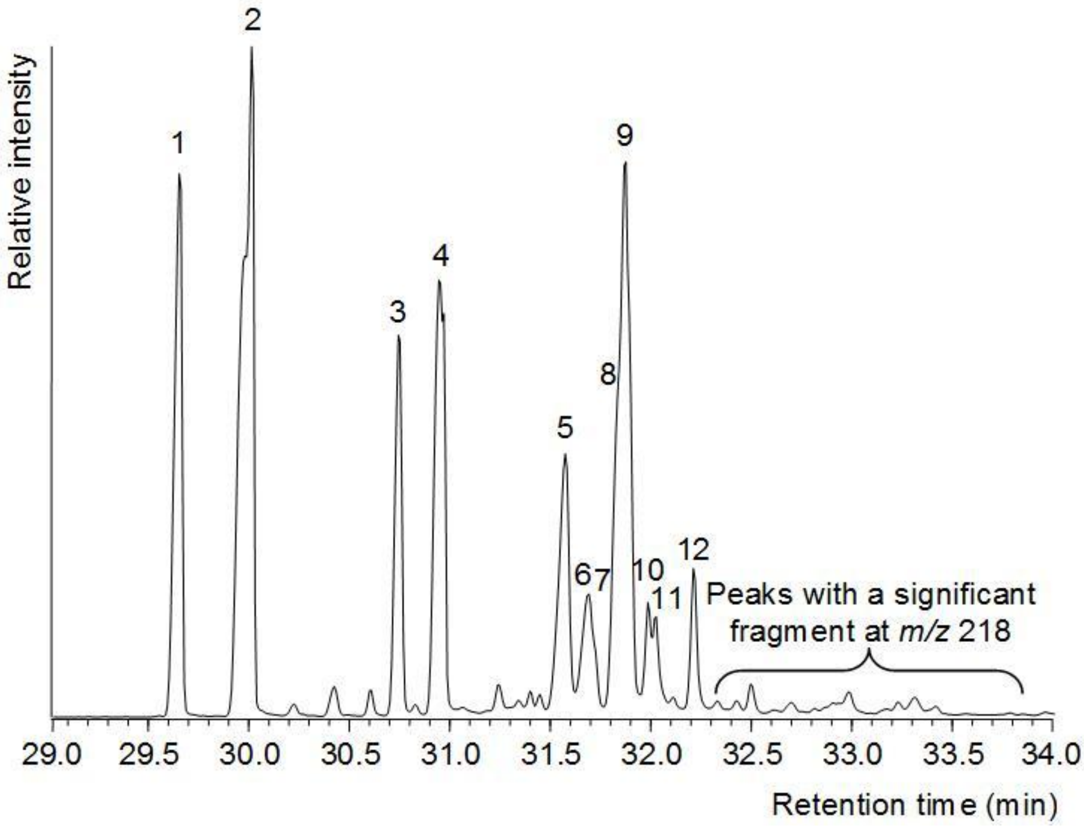


Figure 7.76. XIC (*m/z* 218) triterpenic compounds, amorphous residue (MS1), cremation burial, Mersea Island barrow, Essex, UK. Peak identifiers relate to **Table 7.32**.

Table 7.32. Identification of triterpenic compounds (TMS derivatives), amorphous residue, cremation burial, Mersea Island barrow, Essex, UK and modern *Boswellia carterii* gum-resin used in a degradation experiment based on molecular ion (M^{+}•), base peak (BP) and key fragment ions.

Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
1	394	218	379, 323, 257, 229, 203>189, 175, 161, 147, 135, 119	24-norolean-3,12-diene
2	394	218	379, 341, 281, 203<189, 175, 161, 147, 133, 119, 107	24-norursa-3,12-diene
3	498	218	483, 393, 327, 279, 257, 203>189, 175, 147, 121	3-epi-β-amyrin
4	498	218	483, 408, 393, 229, 203<189/190, 175, 161, 147, 121	3-epi-α-amyrin
5	408	218	393, 353, 273, 255, 232, 203, 189, 161, 135	24-norursa-3,12-dien-11-one
6	424	218	409, 391, 367, 313, 257, 203, 189, 175, 161, 135, 109	β-amyrenone
7	498	218	483, 468, 408, 393, 311, 241, 203, 189, 161, 129, 69	β-amyrin
8	424	218	409, 393, 311, 257, 245, 203, 189, 175, 161, 135, 121	α-amyrenone
9	498	218	483, 468, 408, 393, 279, 257, 203=189, 175, 135, 119	α-amyrin
10	426	218	411, 393, 379, 257, 203=189, 175, 161, 147, 135, 119	unidentified ursane derivative
11	600	218	585, 510, 495, 382, 292, 203, 189, 161, 147, 135, 107	α-boswellic acid
12	600	218	585, 510, 495, 382, 292, 203, 189, 161, 147, 133	β-boswellic acid

Low levels of the precursor α- and β-boswellic acids (**11** and **12**) confirmed this identification. These moieties are biomarkers for *Boswellia* spp. gum-resins (Evershed *et al.* 1997a) although they are often in low abundance in fresh frankincense and may even be absent from some *Boswellia* species' exudates (Başar 2005, 132-6, 147-50). Additional corroboration was provided by the presence of incensol, verticilla-4(20),7,11-triene and related diterpenes (**Figure 7.77**). This combination of di- and triterpenic compounds appears to be limited to certain members of the genus *Boswellia* (Başar 2005: 41-96; Hamm *et al.* 2005). Comparison with modern botanically and geographically-certified *Boswellia* spp. exudates (**Appendix 3.3**) demonstrated that the compounds in the archaeological samples matched those in the reference materials.

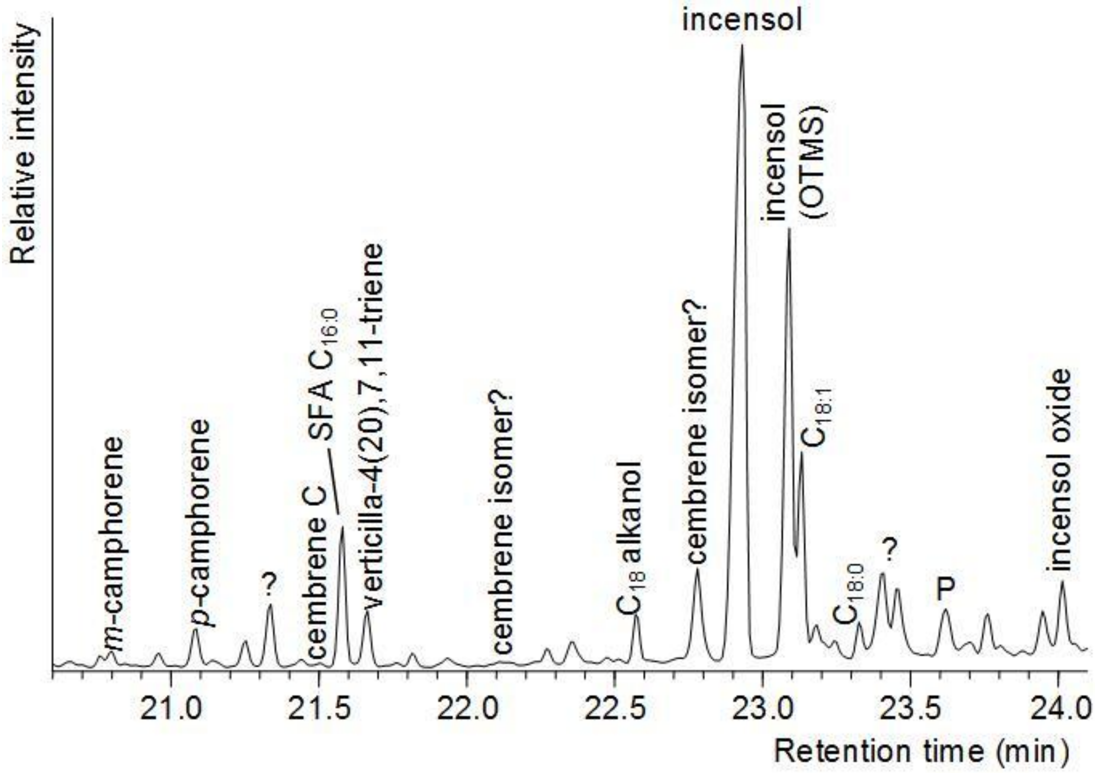


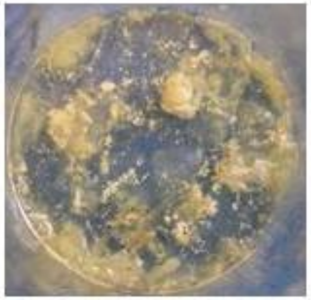
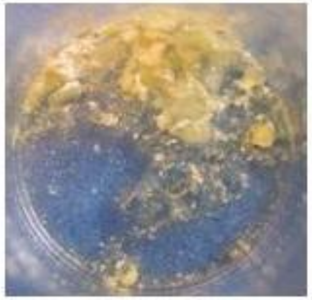
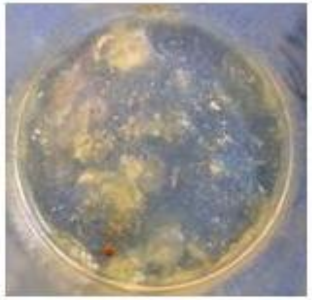
Figure 7.77. Partial TIC (20.6-24.1 min) incensol and related diterpenic compounds, amorphous residue (MS1), cremation burial, Mersea Island barrow, Essex, UK.

Despite this clear identification, the relative abundance of the various compounds varied considerably in relation to the modern gum-resins where the boswellic acids and their *O*-acetyl derivatives were dominant. Indeed, the latter appear to be absent from the Mersea Island samples although traces of HMM moieties with a significant fragment ion at *m/z* 218 but whose molecular ion could not be discerned, were noted. The pattern in the archaeological samples reflects the reduction in triterpenic acid moieties and range of defunctionalised compounds produced by pyrolysis (Başar 2005: 151-184; Mathe *et al.* 2007; van Bergen *et al.* 1997). Nonetheless, the presence of a broad range of highly volatile mono- and sesquiterpenes which would be lost by heating in excess of 50 °C (Hamm *et al.* 2003) and absence of any markers of extensive heating in the Pinaceae fraction, suggests that this resemblance is due to equifinality. This is supported by the fact that nordienes and nortrienes were present in all of the modern exudates whose history precludes deliberate thermal alteration and emphasises the need for further mapping of the impact of environmental factors on the degradation pathways of *Boswellia* spp. gum-resins.

The retention of the mono- and sesquiterpenes is also a matter of interest. These volatile components rarely survive over archaeological time but may have done so here due to the considerable quantity of resinous material deposited within the urn. In addition, as the glass vessel was part filled with a liquid when it was discovered, their retention may have been due to the 'waterlogged', restricted oxygen, microenvironment which had resulted in the formation of the protective white coating. As no evidence for a transformed fat/oil (as recovered from the Purton cremation vessel) was evident, this substance appears to be a mucilage formed by the reaction of the polysaccharide (gum) components and the liquid, although no sugars were observed. The anaerobic/anoxic conditions within the glass vessel might also account for the absence/low abundance of acetyl derivatives.

A simple laboratory experiment was, therefore, conducted to assess the effect of immersion on *Boswellia* spp. exudates. Modern samples (1 g) were placed in vials of water (2 ml at pH 6, pH4, pH2) and wine (white, Ponte Guglie, Italy), since the latter could have been deposited in the urn as a libation, and left to stand for 14 days (room temperature and pressure) (**Table 7.33**). Any excess fluid was drained away and the samples left to air dry (in order to mimic the post-excavation treatment of the Mersea Island materials). A portion of each was analysed by GC-MS alongside an untreated sample of the original gum-resin which had been stored in the laboratory for nine months since initial evaluation upon purchase. Rapid formation of the mucilage was observed in all cases. The extent of this change varied in relation to species (presumably due to differences in their polysaccharide:terpenoid content) rather than pH (**Table 7.33**).

Table 7.33. Images showing changes in modern *Boswellia* spp. gum-resins as the result of immersion in water (at three pH values), wine and water mixed with wine for three days (Author).

	Initial	3 days water pH6	3 days water pH4	3 days water pH2
B. carterii				
B. serrata				
B.carterii **a. after 1 hour in water pH4** **b. after 3 days in white wine** **c. after 3 days in water + wine**				

No significant change in the chemistry of the resin fraction was detected between the untreated reference material and the samples that had been immersed in any of the liquids (**Figure 7.78; Table 7.32).**

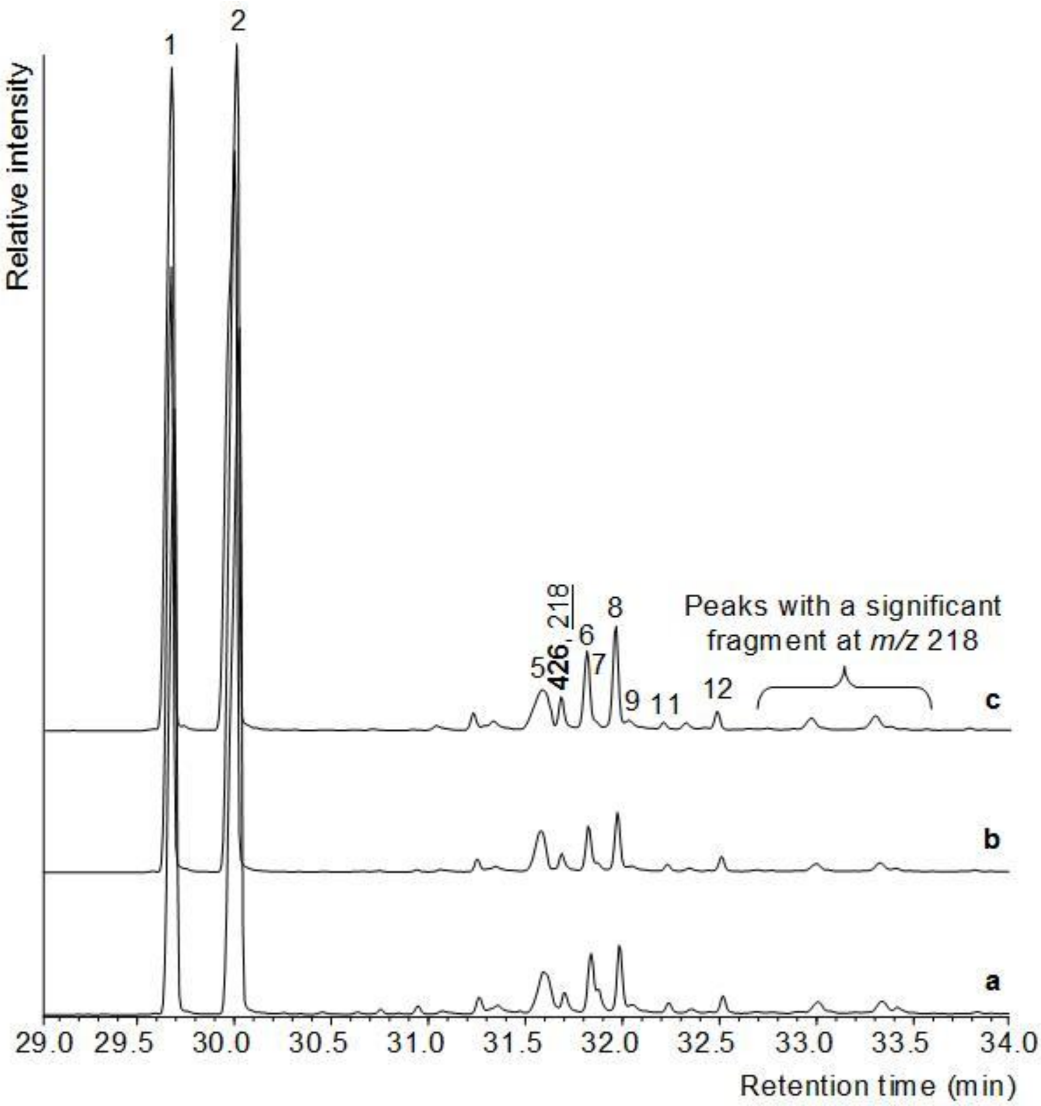


Figure 7.78. XIC (*m/z* 218) triterpenic compounds, modern *Boswellia carterii* gum-resin: a. untreated sample extract; b. after 3 weeks in water, pH 6; c. after 3 weeks in a mixture of water and wine. Peak identifiers relate to **Table 7.32**.

There was, however, an important, if unintended, consequence of this experiment which resulted from the re-analysis of the reference gum-resins. These had originally been characterised soon after purchase (see **Appendix 3.3**). A portion of each was then curated in a glass vial, largely in the absence of light, for nine months although exposure to both light and air would, occasionally, have occurred due to opening of the cupboard, removal of the sample box for research purposes, display/smelling of the samples for teaching purposes and being left to stand on the bench top for a few days prior to re-analysis. Nonetheless, comparison of the results of the initial and repeat analyses of these, otherwise, untreated samples showed a considerable alteration in chemical composition with a dramatic increase in the abundance of the nordienes, concomitant reduction in the resin acids and almost complete loss of their *O*-acetyl derivatives (**Figure 7.78**). Such changes, therefore, clearly imitate those reported in heated (i.e. burnt as incense) *Boswellia* spp. exudates (c.f. Başar 2005: 151-184; Baeten *et al.* 2014; Mathe *et al.* 2007).

7.11.4 Summary and interpretation of results

Analysis of the amorphous residue recovered from a glass vessel containing a 2nd century AD cremation burial interred in the centre of a round barrow on Mersea Island, Essex, UK provided evidence for two different resins. The first was represented by a range of pimarane and abietane-skeleton diterpenoids characteristic of a Pinaceae resin. The far more abundant exudate was identified as frankincense, the gum-resin of member of the genus *Boswellia*. As classical authors regularly issued warnings about the cutting of more valuable products with less expensive resins (*Pliny NH 12.32:65* nd; *Dioscorides 1.81* nd), it is possible that the conifer contribution to the Mersea Island material may be the result of this unscrupulous practice. Nonetheless, although the commonly occurring LMM mono- and sesquiterpenes present could derive from this combination of exudates but there is also a chance (given their range and abundance) that they may represent additional, unidentified, contributions from other resinous species (e.g. essential oils or exudates not containing diagnostic HMM components, **5.2-5.5**). If this was a scented unguent, however, it was not one that was oil/fat based. Moreover, the range of compounds present (LMM mono- and sesquiterpenes; resin acids and their derivatives) indicated that this aromatic mixture had not been burnt on the pyre or as incense prior to deposition.

The considerable amount of resinous matter recovered, in conjunction with the ‘waterlogged’, low oxygen microenvironment within the covered ossuary, may account for the exceptional level of preservation. Thus, the fact that the urn had been about a third full of liquid when discovered prompted a simple laboratory experiment. Results showed that, upon immersion, a 'milky' solution formed leaving a residue of white amorphous masses embedded with orange crystalline fragments. This semi-solid material closely resembled the visual appearance of the archaeological samples collected from the urn. Moreover, the chemical composition of the resin fraction of the curated modern gum-resins assessed was largely unaffected regardless of the nature/pH of the liquid employed. Nonetheless, even after only nine months of storage, the range and relative abundance of the compounds present differed markedly from the original fingerprint ascertained soon after purchase with a significant decrease in the boswellic acids, loss of their *O*-acetyl derivatives and increase in their neutral degradation products. No markers for wine (e.g. syringic acid) were detected in the samples that were left to stand in the white wine/water and wine so, although they were absent from the Mersea Island residues, a contemporaneous libation of wine cannot be precluded.

This discovery of an abundance of frankincense mixed with a lesser quantity of a Pinaceae resin in an elaborate cremation burial below a round barrow on a tidal island off the east coast of Britain is of considerable importance. It provides chemical confirmation for the deposition of resinous exudates in a Roman period cremation burial, seemingly as an unburnt offering, and enhances our understanding of the nature of this rite when observed as part of later Roman mortuary practices. The implications of these findings in relation to the evidence recovered from the cremation burial from Purton and the range of 'package' burials analysed as part of this project are discussed in the following chapter.

Chapter 8

Discussion: resinous substances and ritual action

"Sprinkle my ashes with pure wine and fragrant oil of spikenard; bring balsam to... Unending spring pervades my tearless urn: I have but changed my state, and have not died. I have not lost a single joy of my old life, whether you think that I remember all or none."
Ausonius Epitaph 6.31 'On the tomb of a happy man' nd

8.1 Introduction

For centuries, repeated references have been made by historians and other researchers to the use of aromatic plant products, including resinous exudates, as part of Roman mortuary rites (**1.1**). Such discussions have, primarily, been based on a limited number of passages penned by elite males working in the milieu of Rome around the turn of the first millennium. Mostly poetic in nature, these works allude to the burning of incense but also mention the direct application of exotic, highly valued substances to the body of the deceased. Employed at every stage of the *funus*, they appear to have formed part of anointing oils and unguents, been piled upon the bier and even moulded into effigies which accompanied the dead to the pyre and, perhaps, the grave. In addition, they appear to have been scattered as votive offerings onto the flames or into the tomb (**3.2**).

The relevance of these descriptions to mortuary practices elsewhere in the Roman world, both chronologically and geographically speaking, has been a matter of considerable debate. In recent years, chemical analysis of isolated finds from Italy, Greece and France have given credence to earlier antiquarian observations that the use of resinous substances in the mortuary sphere extended into the provinces (**3.3**). Supported by the detailed examination of intact sarcophagus burials from Trier, Germany (Reifarth 2009, 2013: passim), the contention that resins formed part of a late Roman package which included inhumation in stone sarcophagi and/or lead-lined coffins, high quality textiles and, on occasion, the use of plaster body-casings has gained ground. Connections between this practice and various aspects of the identity of the deceased have been suggested, for example as a mark of their social status or to distinguish followers of Christ (Philpott 1991: 92; Pearce 2013; Ramm 1971; Sparey Green 1977; **3.4**).

These hypotheses have remained unproven, since no systematic study of this burial class from a single province has been undertaken. Given the relatively small number of such ‘package’ burials from Britain, absence of previous chemical confirmation for the use of resins in Roman mortuary contexts and the limited potential for natural environmental contamination (in terms of resin-producing species), this region seemed an appropriate testing ground (**4.5**; **5.6**). Thus, the main purpose of this project was to establish whether or not resinous substances had been used in the treatment of the dead in Roman Britain and, if so, which exudates had been employed. To achieve this aim samples from extant stone sarcophagus, lead-lined coffin and/or plaster burials were analysed (**7.1-7.9**). These materials were selected from both urban and rural burial grounds and were associated with the remains of individuals of both sexes and all ages so that previous conjecture concerning the distribution (i.e. predominantly urban) and significance (i.e. more commonly associated with young females) of this practice could be addressed. Likewise, where possible, comparison with normative inhumations was undertaken to explore the proposed relationship with social status. Residues from elaborate cremation burials from Britain (**7.7; 7.10**), a recent find from Bezannes in northern France (**Appendix 4**) and Egyptian votive mummies (**Appendix 5**) dated to the Ptolemaic-Roman period, were also assessed to supplement data compiled from the literature and illuminate the long-standing debate over the purpose and origins of this rite.

The following discussion will address each of the research questions set out in **1.2** by establishing which findings are of archaeological relevance (**8.3**), the identity and geographical origins of any resinous substances present (**8.4**) and the manner in which they were utilised in order to consider their practical and/or ritual purpose (**8.5**). These analytical findings are then explored for repeated associations with other material aspects of this proposed burial class, compared with pre-Roman Iron Age (PRIA) and normative rites in Roman Britain and placed in the broader context of mortuary practices across the Empire to address arguments about their relationship with aspects of the deceased’s social persona (**8.6**). Finally, the meaning of this mid-late Roman body treatment will be evaluated within the framework of mortuary theory as outlined in **Chapter 2** focussing on the agency of materials (**8.7**). First, however, additional aspects of significance in terms of the collection and retention of samples will be addressed (**8.2**).

8.2 Organic residue analysis and the value of dirt

The decay of organic matter is part of the natural order of things so seeking to access such evidence in the archaeological record is, in essence, a battle against the odds. Vast losses as a result of taphonomic processes are to be expected. Nonetheless, as decades of research have demonstrated, a surprising amount of information may be retained even in the most unprepossessing of samples (c.f. Colombini & Modugno 2009; Evershed 2008a; Pollard & Heron 2008: 235-269). During this project, when dealing with residues from mortuary contexts additional considerations were found to have impacted on the survival of suitable materials. These included attitudes towards the appropriate treatment of human remains which had, prior to 1950s, often resulted in their reburial soon after discovery (e.g. Cirencester, Dartford, York). Thus, only artefacts such as jewellery, glassware and coins were retained and, on occasion, sarcophagi, lead-liners and plaster body-casings, with precise provenance rarely recorded. Activities such as the open-air display of sarcophagi, loss or theft of lead-liners and retention of crania, often in private collections, were also encountered (e.g. Dorchester, London, York).

Likewise, financial constraints placed on archaeology in more recent years were found to be a factor resulting in reburial of sarcophagi without excavation (e.g. Northover House site, Somerset; Minnett 2013, pers. comm., 08 November), restricted post-excavation analysis and difficulties in maintaining long-term storage in museum collections. Standard practices such as the cleaning of human remains for osteological analysis and the sieving/flotation of grave deposits, frequently using industrial methylated spirits, with bulk discard of the organic matter also created problems alongside less common treatments such as the storage of grave deposits in containers filled with water. Consequently, many of the materials sought were no longer extant while others had been ear-marked for disposal including the plaster from Poundbury and ‘environmental’ samples (grave deposits) associated with the ‘Spitalfields Lady’, London.

That anyone would wish to access these materials for evidence of the treatment of the body was often met with surprise. Indeed, the concept that invisible traces of archaeological significance could be retained within samples that were viewed as ‘dirt’ was, for the most part, unfamiliar. This was largely due to misconceptions concerning the scope of organic residue (lipid) analysis, the most common being that this technique could only be used to evaluate issues regarding diet through the analysis of samples from ceramics. Questions asked prior to or during sampling sessions and in response to podium and poster presentations revealed that many people were unacquainted with the fact that resins were natural products from trees and shrubs which contained compounds accessible by gas chromatography-mass spectrometry (GC-MS). That terpenoids formed a sub-class of lipids was also queried since lipids were commonly conceived to comprise components of fats, oils and waxes (e.g. carboxylic acids, wax esters, sterols). Moreover, scientific research related to mortuary contexts that did not involve looking at inorganic artefacts created confusion with other techniques (e.g. isotope analysis) leading to the assumption that destruction of portions of the human remains would be required. Thus, despite its long-standing utilisation in a wide variety of archaeological and forensic contexts (e.g. Bethell *et al.* 1994; Bull *et al.* 2000, 2009; Connan 2012: passim; Evershed *et al.* 1997b; Forbes *et al.* 2003), awareness of the broader applications of organic residue analysis does not appear to be widespread among excavators, curators, conservators, academics in related disciplines (e.g. chemistry, anatomy, mass spectrometry) or, indeed, our own.

It is clear, therefore, that organic residue analysts need to finds ways to engage with a wider audience and enhance the profile of the broader applications of the technique in the face of the popularity of more direct methods of addressing human lifeways (e.g. isotopes, DNA, proteomics). In terms of the mortuary sphere, guidance regarding the collection of suitable samples would seem to be a good starting point, based on concerns expressed by excavators and museum staff. At present there appears, perforce, to be an all (fear of criticism for not retaining materials of future interest) or nothing (lack of storage space and funding for analysis) approach. This needs to change for the benefit of everyone, since the success of the current project rested on its systematic targeting not only of visible residues but also of the hitherto disregarded ‘dirt’ from the base of the more substantial burial containers (and ‘mummy dust’, the inevitable losses of small fragments from mummy bundles over time, **Appendix 5**). This largely untapped reservoir of chemical evidence has the potential to retain biomarkers, diagnostic of resinous substances employed in the treatment of the body, long after the decomposition of macro-scale organic materials. The case of the ‘Spitalfields Lady’, London (**7.4**) illustrates this point as insufficient evidence remained in the residue from the hyoid bone to clarify the presence of one resin in this burial whereas the grave deposits provided definitive confirmation that two exudates had been used in the treatment of the body.

Thus, in mortuary contexts, it is recommended that samples need not be collected from normative inhumations unless exceptional circumstances prevail (e.g. formation of adipocere, extensive adhering residues, plaster body-casings) as taphonomic factors generally preclude a successful outcome. From stone sarcophagus and lead-lined coffin burials, ten to twenty spatially distributed <5 g (a full scintillation vial) samples from around the skeletal remains, with at least one control, should be collected (**Appendix 2.2**). It should be noted, however, that where these more substantial containers demonstrate extensive soil and/or water ingress, such sampling has not been productive (e.g. Somerset, Leicestershire). With regards to cremation burials, unusual organic matter (e.g. amorphous masses) and related materials (e.g. liquids) should be retained. It is hoped that guidelines of this nature will promote the collection and retention of suitable samples from mortuary contexts prior to the processing of grave deposits. A new volume of advice about the potential of organic residue analysis, compiled by Dr Julie Dunne, University of Bristol (with a contribution from the author) should assist in this endeavour (Historic England 2016; **Appendix 7.5**).

Table 8.1. Burials found to contain resinous substances. **N.B.** age determinations are those given in the relevant publications; LSC = lead substituted carbonate.

Location	Date (c. AD)	Age (years)	Sex	Body position	Container(s) + *fill*	Associated finds	Nature of sample	Resinous exudate Evidence
Arrington infant Wraggs Farm, Arrington, Cambridgeshire	2nd-3rd	c. 1	--	extended, supine, head to west	wood with lead-liner *traces of plaster (LSC)*	Pipeclay figurines (in box on foot end;, dyed-wool fragments; hair	Amorphous orange fragments	*Pistacia* spp. Resin acids + derivatives
'Spitalfields Lady' 280 Bishopsgate & the Spitalfields ramp, London, E1 SRP98, Site K, SK15903	4th	20-25	F	extended, supine, head to west	limestone sarcophagus decorated inner lead coffin	Textiles (wool, silk); gold thread; 'pillow' of bay leaves; jet and glass items	Residue adhering to hyoid; grave deposits from base of lead coffin and between coffin and sarcophagus	Pinaceae + *Pistacia* spp. Resin acids + derivatives
Eagle Hotel site, Andover Road, Winchester, Burial G336	4th	35-45	M	extended, supine, NS aligned	wood with lead-liner	Mineralised textiles; coin of Constantine	Grave deposits from base of lead-liner	Pinaceae + ?*L. orientalis* Phenolics + resin acids
Northview Hospital, Purton, Wiltshire, Grave 1	4th	18-25	F	extended, supine	stone sarcophagus lead-liner *traces of plaster?*	Glass and ceramic vessels; shale bracelet; animal bones; textiles, wool/dyed-border	Residues/debris associated with skeletal elements and lead fragments	Pinaceae + *Pistacia* spp. Resin acids + derivatives
Northview Hospital, Purton, Wiltshire, Grave 2	?	25-40	?F	cremation burial	limestone ossuary cylindrical lead urn glass vessel	Cremated bird and animal bones; fragments of burnt ceramic; charcoal	Amorphous residue from surface of liquid within glass cremation vessel	?*Boswellia* spp. + ?other resin(s) Triterpenols + neutral derivatives
Poundbury, Dorchester, Dorset R2 mausoleum Grave 8	4th	--	--	extended, supine, head to west	limestone sarcophagus *gypsum body-casing*	Textile impressions; fragments of bone comb	Residues adhering to inner surface of body-casing	Pinaceae Resin acids + derivatives
Poundbury, Dorchester, Dorset Site E Grave 127	4th	15	F	extended, supine head to west	wood with lead-liner *traces of plaster (LSC)*	Textile impressions	Residues adhering to mineralised textiles	Pinaceae Neutral derivatives
Poundbury, Dorchester, Dorset R10 mausoleum Grave 517	4th	35	F	extended, supine head to west	limestone sarcophagus *gypsum body-casing*	Textile impressions; bone comb; copper alloy ring	Residues adhering to inner surface of body-casing	Pinaceae Neutral derivatives
Poundbury, Dorchester, Dorset R9 mausoleum Grave 529	4th	40	F	extended, supine head to west	wood with lead-liner *plaster body-casing*	Textile impressions; hair	Residues adhering to inner surface of body-casing	Pinaceae Neutral derivatives
Poundbury, Dorchester, Dorset R9 mausoleum Grave 530	4th	30	M	extended, supine head to west	wood with lead-liner *gypsum body-casing*	Textile impressions; hair [Wool band, with organic residue; Reifarth 2013]	Residues adhering to inner surface of body-casing	Pinaceae Neutral derivatives [Balsam - resin acids]

Poundbury, Dorchester, Dorset Site E Grave 892	4^{th}	40	M	extended, supine	wood with lead-liner *traces of plaster*	Textile impressions	Residues adhering to mineralised textiles	Pinaceae DHA + suite of derivatives
Poundbury, Dorchester, Dorset Site E Grave 1040	4^{th}	30	M	extended, supine	wood with lead-liner *plaster body-casing (LSC)*	Textile impressions	Residue adhering to mineralised textiles	Pinaceae DHA + suite of derivatives
Alington Avenue, Dorchester, Dorset, Grave 3664 SF 1075	3^{rd}	45+	F	extended, supine, head to SW	Wood lid of sandstone *chalk rubble packing*	Hobnailed footwear; glass vessels; ?food offerings	Grave deposits associated with cranium	*Boswellia* spp. Resin acids + derivatives
Alington Avenue, Dorchester, Dorset, Grave 4378 SF 1169	3^{rd} C	4-6	?	extended, supine, head to SE	wood with lead-liner *chalk environmental ingress*	Ceramic jar; coin; iron rod; garment with purple dyed stripes on shoulders	Grave deposits and residues adhering to skeletal elements	*Boswellia* spp. Resin acids + derivatives
Railway excavations, York YORYM: 2007.6206	--	--	--	--	stone cist cedar wood coffin *plaster body-casing*	Fragments of coarse textile	Residue adhering to inner surface of body-casing	*?Boswellia* spp. Triterpenols + neutral derivatives
Mill Mount, York YORYM: 2007.6205i	--	--	--	--	-- *plaster body-casing*	Textile impressions	Residue adhering to inner surface of body-casing	*?Boswellia* spp. Triterpenols + neutral derivatives
Railway excavations, York YORYM: 2010.1219	--	adult	--	--	stone sarcophagus *plaster body-casing*	--	Grave deposits from base of sarcophagus	*Pistacia* spp. Resin acids + derivatives
Mersea Island barrow (Mersea Mount), Mersea Island, Essex	2^{nd} C	35-45	M	cremation burial	tile-built chamber square lead ossuary glass vessel	--	Amorphous yellow-white masses enclosing orange fragments	Pinaceae + *Boswellia* spp. Resin acids + derivatives

8.3 Evidence for substances of archaeological relevance

Results obtained from the analysis of amorphous masses (#3 burials), adhering/adsorbed residues (#12 burials) and/or grave deposits (#6 burials) provided evidence for the presence of resinous plant exudates in sixteen inhumation and two cremation burials (**Table 8.1**). Those samples that were found to contain suites of biomarkers including precursor resin acids and/or diagnostic derivatives permitted secure identification of botanical source (#10 inhumations; Mersea Island cremation). Where only traces of pentacyclic alcohols, their derivatives and defunctionalised terpenic compounds were present (#6 inhumation burials; cremation, Grave 2, Purton) characterisation was less certain. Indeed, of themselves, some of these finds would not have been considered sufficient evidence for the inclusion of resinous substances (e.g. #4 from Poundbury). Given the absence of terpenic compounds in the controls (where available) and the weight of definitive proof these may, however, also be considered indicative of the use of resinous substances. This contention is supported by the fact that these degraded compounds represent examples of the same, limited number of, plant exudates recovered from the other burials (**8.4**). Moreover, most of the samples from which they were obtained had been exposed to extensive oxidation as they came from plaster body-casings that had been loosely wrapped in plastic sheeting or simply placed on a layer of foam during long-term storage.

Residues from a further three burials from York (YORYM:2007.6214, 2010.1201, 2010.1196), similarly, contained traces of terpenic compounds. Evidence of petroleum products (which may contain terpenes if derived from terrestrial sources) were, however, also present with hopanes observed in samples from two other York plaster body-casings (YORYM:2007.6213, 2007.6210). Theoretically, the fossil hydrocarbon contribution to these finds as in samples from Context 1, Armagh Road, London (again in conjunction with triterpenic compounds), could indicate the application of bitumen as a preservative, as reported in some Egyptian mummies (c.f. Colombini *et al.* 2000; Connan *et al.* 1992; Harrell & Lewan 2002; Maurer *et al.* 2002). Nonetheless, the presence of modern contaminants in the form of pesticides (which may be dispersed in petrol) in the York samples and the discovery of the Armagh Road sarcophagus during construction work suggest that this data must be discounted at present. Thus, in addition to the resinous substances securely identified, only the fatty matter from the cremation burial (Grave 2), Purton should (due to the closed context and curational history of the samples, **7.7**) be considered of archaeological relevance in terms of materialised ritual actions associated with the remains of the deceased

8.4 Botanical sources and geographical origins

The suites of terpenic compounds observed in the sixteen stone sarcophagus and/or lead-lined coffin burials and two cremation burials that produced positive results provided evidence for four resinous exudates of archaeological interest. In most cases, only higher molecular mass (HMM) di- and triterpenoids have survived although, in exceptional circumstances (e.g. Mersea Island barrow cremation), low molecular mass (LMM) terpenes and/or phenolics were observed. Given the chemical composition of many aromatic exudates, as discussed in **Chapter 5**, the impact of natural taphonomic processes (e.g. evaporation, leaching) and post-discovery anthropogenic choices (detailed above, **8.2**), it is almost certain that traces of other scented substances have been lost. Nonetheless, these finds were able to end centuries of speculation concerning the use of resins and gum-resins as part of Roman mortuary rites in Britain and extend this knowledge to establish which exudates were employed.

The first was found to be a diterpenoid resin obtained from members of the Pinaceae family, as denoted by precursor acids with abietane and pimarane skeletons (e.g. pimaric, *iso*pimaric, abietic acids) and their derivatives (e.g. dehydroabietic acid, didehydroabietic acid, methyl dehydroabietate) (**5.3.2**; **Table 5.3**). This conifer resin was identified in seven plaster burials from Poundbury (**7.8**) and, in conjunction with other resinous substances, as part of the mortuary rites accorded the ‘Spitalfields Lady’, London (**7.4**), the focal burial of a mature adult male from the Eagle Hotel site, Winchester (**7.5**), a young adult female interred in a rural burial ground at Purton, Wiltshire (**7.7**) and the cremated remains of a mature male placed at the centre of the Mersea Island barrow (**7.11**). When combined with other exudates, the Pinaceae contribution appeared to be the minor component.

All members of the Pinaceae, a family of gymnosperms which includes pines, firs and larches, produce resins to some degree (*Dioscorides 1.92* nd; Howes 1949: 106-110; Langenheim 2003: 319-322). The similarity in their chemical composition, however, generally prevents clear differentiation even in modern materials (Mills & White 1999: 99). In the archaeological record, the homogeneity produced by taphonomic changes adds to the unreliability of any attempt at a lower taxonomic determination (Colombini & Modugno 2009). Nonetheless, in this instance, *Abies* spp. (firs) and *Larix* spp. (larches) appear less credible candidates due to the absence of neutral labdane compounds in these samples and minimal evidence for the exploitation of these genera, alongside *Picea* spp. (spruces), during the Roman period. Likewise, the wood and essential oils of *Cedrus* spp. appear to have been utilised rather than their resin fraction. For example, remnants of the coffin which contained plaster burial YORYM:2007.6206 from the railway excavations, York were identified as cedar. This is supported by traces of sesquiterpenes consistent with Pinaceae or Cupressaceae products in a residue from a crack (possibly created by a coffin nail) in the plaster.

Moreover, the best preserved examples of Pinaceae resins from the burials analysed (e.g. 'Spitalfields Lady', London; Grave 8, Poundbury; Mersea Island barrow cremation) contained an abundance of pimaric and abietic acid isomers and their degradation products. This combination is considered more characteristic of *Pinus* spp. (pine) resins (Mills & White 1977; Colombini *et al.* 2000) as confirmed by evaluation of a range of reference materials (**Appendix 3.1**). As this genus is the both largest and best attested in terms of its exploitation in antiquity, with species such as *Pinus pinaster* (Maritime pine) and *Pinus halepensis* (Aleppo pine) the most abundant natural resin producers within the Pinaceae family (Howes 1949: 106-110; Langenheim 2003: 319-320), these finds probably represent pine resins although certainty is not possible on this point.

Likewise, the geographical origin of these exudates cannot be definitively established as members of the Pinaceae are widespread in the northern hemisphere. Given the distribution (northern highlands largely beyond the boundary of the province) and relatively low resin production of the only native British Pinaceae species, *P. sylvestris*, however, it seems likely that these conifer resins were imported during the Roman period. There is, in fact, evidence from PRIA Gaul, that southern France was a centre of resin production. Largely harvested from *Pinus* spp., this industry continued to flourish after the conquest in both France and Spain with long-term exploitation of this natural resource also attested in Italy and Greece (Howes 1949: 106-110; Langenheim 2003: 319-321; Stern *et al.* 2008a). Thus, although traces of Pinaceae resins in *amphorae* or on other ceramic vessels are generally interpreted as sealants, based on this new information regarding their use as part of mortuary practices across Roman Britain it seems that they may also have been imported as valuable commodities in their own right. This trade in Pinaceae products is supported by evidence for the importation of pine pitch from the Mediterranean (Evershed 1993) and by isotope ratios, indicative of origins in warmer climes, obtained from pine resins coating both imported and locally produced ceramic vessels from Roman London and Carlisle (Heron & Pollard 1988; Stern *et al.* 2008a).

The second exudate derived from further afield and was denoted by a characteristic suite of triterpenoids including moronic, oleanonic, masticadienonic and *iso*masticadienonic acid and their derivatives. These compounds are biomarkers for mastic/terebinth resins obtained from members of the genus *Pistacia* (Modugno & Ribechini 2009; Papageorgiou *et al.* 1997; Serpico & White 2000). One of the most readily identifiable resins in the archaeological record, this combination of precursor acids was observed in visible orange fragments recovered from a lead-lined coffin burial at Arrington, Cambridgeshire (**7.2**). These amorphous materials, found around the cranium of a year-old infant, comprised a pure *Pistacia* spp. resin. Combined with evidence for Pinaceae resins, these diagnostic compounds were also observed in grave deposits associated with two young adult females, the ‘Spitalfields Lady’, London (**7.4**) and Grave 1, Purton, Wiltshire

(**7.7**). In addition, traces of 28-norolean-17-en-3-one and what appears to be oleanonic acid (alongside unrelated triterpenes, see below) were present in the cremation burial (Grave 2) from Purton (**7.7**) although, this is insufficient proof of the inclusion of mastic/terebinth. The range of oleanane derivatives (e.g. 28-norolean-12-en-3-one; 28-norolean-17-en-3-one), dominated by oleanonic aldehyde, and trace of oleanonic acid recovered from in situ grave deposits from a plaster burial (YORYM: 2010.1219) found during the railway excavations at York (**7.10**), however, strongly indicate the presence of a *Pistacia* spp. exudate.

These findings are supported by evaluation of a range of modern *Pistacia* spp. resins (**Appendix 3.2**). This corpus of data illustrates the considerable natural variability in the chemistry of this genus which may be due to growing conditions and/or the differential impact of post-harvest histories, as reported in the literature (e.g. Assimopoulou & Papageorgiou 2005a, 2005b). One notable aspect is the absence or low abundance of the resin acids, particularly the tirucallanes, in some of the modern reference materials analysed (e.g. *P. khinjuk*, Iran). This is best illustrated through comparison of the two *P. terebinthus* exudates (from Cyprus and Iran) which were both visibly (**Table 6.1**) and chemically different (**Appendix 3.2**). Indeed, the former, despite appearing the better preserved (translucent, orange/yellow, slight scent remaining) and having been curated for a shorter length of time, contained only highly degraded derivatives which would not have permitted secure identification as *Pistacia* spp. in the archaeological record. This is especially telling as it is the resin acids that cannot be traced (or only with difficulty) in a number of these Roman period burials.

With regards to the archaeological materials (and, perhaps, these *P. terebinthus* reference resins) this reduction in the resin acids may, of course, be the result of differential taphonomic change over time. This factor does not appear, however, to account for composition of the recently purchased *P. khinjuk* exudate and is also contra-indicated by the considerable abundance of the monoterpenes (the more volatile and readily lost components) in these seemingly degraded modern samples. In contrast, both of the relatively fresh

P. lentiscus exudates from Greece were dominated by the classic quartet of resin acids with minimal amounts of monoterpenes evident. These observations hold considerable significance for the recognition and, indeed, survival of evidence for some *Pistacia* spp. resins in archaeological contexts (discussed further in **8.5**). They also impact on questions of species determination with, in this instance, only the suite of compounds in the fragments found with the Arrington infant able to permit any such attempt. Here the presence of less commonly observed components such as 3,4-seco-28-norolean-12(18)-en-3-oic acid and olean-18-enolic aldehyde suggest that the source is more likely to be *P. terebinthus* (or a closely related species) rather than *P. lentiscus*, although this cannot be confirmed.

As members of the genus *Pistacia* have a relatively limited geographic distribution (Zohary 1952) these resins must have been imported into Britain from the Mediterranean or the Levant. Indeed, classical sources provide a considerable body of evidence detailing the exploitation of *Pistacia* spp. exudates focussed on the island of Chios (mastic) and groves in the vicinity of Damascus, Syria (terebinth) (*Dioscorides 1:89-91* nd; *Pliny NH 12.36, 13.12, 14.25, 16:22* nd; *Theophrastus 9.2:2* nd). Trade from these regions with Egypt is also well-attested as a result of the research undertaken by Loret (1949) into the meaning of the term *snṯr* and the chemical analysis of residues from ceramics and mortuary contexts (e.g. Buckley & Evershed 2001; Colombini *et al.* 2000; Mills & White 1989; Serpico & White 2000, 2001; Vieillescazes & Coen 1993). Likewise, evidence for *Pistacia* spp. resins has been obtained from an Etruscan tomb in Italy (in an ointment jar mixed with Pinaceae resin and vegetable oil, Colombini *et al.* 2009) and from a number of Roman period inhumation burials in continental Europe (Bruni & Guglielmi 2005, 2014; Devièse 2008: 63-65; Reifarth 2013: 108-110; **3.3**). The addition of these finds from the remote province of *Britannia* highlights, therefore, the considerable importance of *Pistacia* spp. exudates in antiquity.

The third resinous substance identified was a most unexpected find, given the previously limited chemical evidence for its presence in the archaeological record (**5.4**). Traces of this exotic exudate were represented by a series of compounds with a base peak at *m/z* 218 and key fragments ions at *m/z* 203 and 189 in residues from two plaster burials from York (YORYM:2007.6205i; YORKM:6206; **7.10**) and in the amorphous matter from Grave 2, Purton (**7.7**). These mass spectra are characteristic of the triterpenic alcohols (β- and α-amyrin, their epimers and oxidation products) and triterpenes with olean-12-ene and urs-12-ene skeletons that have a methyl group at C-17. This combination, with a greater abundance of the ursane isomers, suggests a member of the Burseraceae (c.f. Stacey *et al.* 2006). Lower taxonomic classification was hampered by an absence of precursor resin acids. Nonetheless, comparison with the published literature indicated that this sequence was a close match for degraded gum-resins from the genus *Boswellia*, better known as frankincense with the neutral 24-nordienes and 24-nortrienes present considered diagnostic derivatives of the boswellic acids (c.f. Baeten *et al.* 2014; Modugno *et al.* 2006; ten Haven *et al.* 1992).

This relationship was supported by the analysis of a range of modern *Boswellia* spp. reference resins as these displayed a similar array of degradation products (**Appendix 3.3**) and the recovery of matching data from two late Roman inhumations, that of an adult female and a child found at Alington Avenue, Dorchester (**7.8**). Indeed, traces of the boswellic acids, themselves, were present in the latter. This intact lead-lined coffin burial also provided a more secure context when considering the potential for contamination as the grave deposits sampled had simply been collected during excavation (from the base of the coffin) and retained, untreated, in plastic bags. With regards to the York finds, with their far longer and less well-documented history, the reported discovery of frankincense in a recently excavated lead-lined coffin burial from Hungate (Keeley 2013, pers. comm., 12-13 December) lends considerable weight to the argument that they too represent residues of archaeological relevance. Finally, the abundance of amorphous matter recovered from the 2nd century AD Mersea Island barrow cremation (**7.11**) contained a whole gamut of mono-, sesqui-, di- and triterpenes with the HMM compounds (cembrenes, incensol, boswellic acids and derivatives) characteristic of *Boswellia* spp. exudates accompanied by a

Pinaceae resin. There can, therefore, be no doubt that frankincense was employed in a variety of mortuary contexts across Roman Britain.

As ever, identification to species presented considerable difficulties as the majority of the triterpenic compounds identified appear common to degraded and/or heated frankincense from all of the main *Boswellia* species. Comparison with the reported chemical composition of various *Boswellia* spp. gum-resins, however, provided some indications (**Table 5.7; Table 8.2**). For example, the absence of lupane-skeleton triterpenes and abundance of boswellic acid derivatives in all of the samples analysed appears to rule out the distinctive *B. freanana* (Somalia). Thus, the York and Alington Avenue finds could derive from any of the other members of the genus. In relation to the frankincense from the Mersea Island barrow, based on the reference materials sampled for this project, it seems more similar to gum-resins from *B. carterii* (eastern Africa) and *B. serrata* (India/Sudan) rather than *B. sacra* (Arabia) due to the near absence of diterpenes in the latter (**Appendix 3.3**). The presence of camphorenes may support this view as, to date, these compounds have only been reported in *B. serrata* (Başar 2005: 67-81). Nonetheless, taxonomic confusion, natural variability, harvesting methods and the impact of taphonomic processes (as with *Pistacia* spp.) mean that this cannot be confirmed especially as cembrene-skeleton diterpenes have previously been reported in many *Boswellia* spp. gum-resins (**Table 7.25**).

Table 8.2. Comparison of the range of terpenic biomarkers indicative of the presence of *Boswellia* spp. gum-resins in the samples obtained from the Roman period burials from Britain analysed for this project.

Name of compound	**York** both	**Purton** Grave 2	**A. Ave** G3664	**A. Ave** G4378	**Mersea**
diterpenes (cembrenes, incensol + derivatives)					X
24-norolean-3,9(11),12-triene					X
24-norursa-3,9(11),12-triene					X
24-norolean-3,12-diene	X	X	X	X	X
24-norursa-3,12-diene	MAIN	MAIN	MAIN	MAIN	MAIN
3-*epi*-β-amyrin	X	X	X	X	X
3-*epi*-α-amyrin	X	X	X	X	X
24-norursa-3,12-dien-11-one	X	X	X	X	X
β-amyrenone	trace		trace	trace	X
β-amyrin					X
α-amyrenone	X	X	X	X	X
α-amyrin	X	X	X	X	X
α-boswellic acid				X	X
β-boswellic acid				X	X
oxo + acetyl boswellic acid derivatives					traces

The fourth array of triterpenoids again consisted of traces of the pentacyclic alcohols (α- and β-amyrin) but accompanied by a limited number of functionalised compounds with olean-12-ene and urs-12-ene skeletons (based on their characteristic fragmentation patterns) and a base peak at *m/z* 203. These were accompanied by phenolic components identified as benzoic acid, vanillin, cinnamic acid, 3-hydroxybenzoic acid and vanillic acid. Present in solvent extracts from mineral-replaced textiles and lead fragments from the base of the focal burial of a mature adult male, in a lead-lined coffin, Eagle Hotel site, Winchester, this combination indicated the presence of a balsamic resin (**7.5**). A lack of clarity regarding the precise identity of the triterpenoid epimers observed was resolved through the purchase, synthesis and analysis of standards. Evaluation of this data revealed that the compounds in the archaeological materials comprised the resin acids, 3α-*epi*oleanolic, oleanonic, 3β-oleanolic, ursonic and 3β-ursolic, respectively, with the oleananes dominant (**Figure 7.32**).

Despite this research, comparison with the literature and characterisation of a range of resinous substances including various *L. orientalis* exudates and the solvent extract of pulverised *S. officinalis* twigs (**7.5.4.3**; **Appendix 3.4**), an exact match proved elusive. Thus, although many potential sources such as mastic/terebinth, frankincense and myrrh could be ruled out, the use of a resin from an extinct species or one no longer considered a viable producer could not. Likewise, a mixture of botanical extracts remains a possibility since the phenolic compounds might have derived from other sources such as essential oils (e.g. of cinnamon/cassia) or even beeswax (c.f. Leela 2008; Pournou 2008; Regert *et al.* 2001). Nonetheless, the presence of a single resinous exudate is supported by the similar array of triterpenoids obtained from the analysis of residues impregnated into textiles (probably a headband) from Grave 530, Poundbury, Dorchester and number of the sarcophagus burials from Trier (Reifarth 2013: 96-99). Indeed, the latter also contained benzoates and cinnamates indicative of a balsamic resin.

If the compounds in these burials do, indeed, derive from a single source then the strongest candidate is *L. orientalis* whose fresh extracts contain such phenolics and a limited number of triterpenoids with oleanane and ursane skeletons, dominated by the former. This suite has been shown to comprise 3α-*epi*oleanolic, 3α-*epi*ursolic, oleanonic and ursonic acid (alongside 3-*epi*-α- and β-amyrin) so, the main difference between the modern and archaeological materials would appear to lie in the configuration of the -OH group at C-3. The most parsimonious explanation seems, therefore, to be that interactions in the burial environment (possibly with yeast or bacteria), catalysed by the presence of metal ions (abundant in these enclosed contexts due to breakdown of the lead-liners, plaster body-casings, skeletal elements), resulted in epimerisation of the resin acids (and triterpenols). This may have occurred via an equilibrium reaction involving an Oppenauer oxidation and Meerwein-Ponndorf-Verley (MPV) reduction, as suggested by Dr W. Martin (**7.5**). In the Roman period (as today), this storax resin would probably have been obtained from Anatolia (western Turkey) or the Levant (Howes 1950).

8.5 Condition and purpose: taphonomic and anthropogenic factors

Based upon the analysis of visible fragments or residues, these exudates had previously been identified (Pinaceae, *Pistacia* spp., *Boswellia* spp.) or proposed (a balsam) in thirty six Roman period inhumation burials in Europe, all of which had been interred in stone sarcophagi (with the exception of sandarac(?) in the multiple burials in the catacombs of *Santi Marcellino e Pietro*, Rome; **3.3**; **Table 3.4**). What this project has now established is that chemical evidence may remain in a greater variety of sample types including in residues adhering to plaster body-casings (e.g. Poundbury), as invisible traces adsorbed into mineralised textiles and re-deposited lead (e.g. Eagle Hotel, Winchester) and as part of the comminuted materials comprising grave deposits (e.g. Alington Avenue) (**8.3**). Moreover, these samples came, not only from sarcophagi (#5), but also from lead-lined wooden coffins (#8), a wooden coffin with a stone lid (#1, where the surrounding environment was chalk) and plaster body-casings from a wooden coffin in a stone cist (#1) and another where the form of container was unknown (#1) (**Table 8.1**). Thus, although all of these burials were more elaborate than the norm (discussed in

8.6), evaluation of a wider range of samples has demonstrated the broader application of this body treatment, at least in Roman Britain.

8.5.1 Natural degradation

Despite these findings, numerous samples of the same type from similar contexts, alongside all of those from less well-protected burials provided negative results. This raises the fraught issue of how to distinguish between true absence and loss of evidence due to taphonomic factors. Were resinous substances not used in these burials? Or are they no longer traceable in the archaeological record? In the sarcophagi from Trier (#4) that contained only indications of fats/oils (using GC-MS, Reifarth 2013: 96-99) and in the case of the intact, undisturbed double burial from Boscombe Down (where animal skin footwear survived, **7.6**), the lack of terpenic compounds seems to indicate an absence of resinous substances. Nonetheless, if essential oils with their volatile compounds or myrrh with its readily lost sugars (water-soluble) and furanosesquiterpenes (volatile) or exudates from species such as *P. khinjuk* and *B. freanana* (which appear to be almost devoid of the characteristic triterpenic markers found in other members of their genera, **8.4**) had been selected then even in these secure contexts the presence of aromatic plant products might be unidentifiable. Add to this the time involved in the transportation of these luxury goods, sorting and storage at various points in their journey and the significant change in chemical composition (after only nine months) of the modern *Boswellia* spp. reference materials re-analysed as part of the degradation study (**7.11.3.3**) then it becomes clear that absence of evidence is not necessarily evidence of absence.

In addition, based on the data compiled here, it appears impossible to predict with any accuracy when HMM terpenic traces may be present through evaluation of the state of preservation of associated materials. Thus, in the inhumation burials (from Britain and continental Europe) found to contain resinous substances, the condition of the skeletal remains varied from very poor (e.g. Trier) to excellent (e.g. 'Spitalfields Lady'), plaster was present in abundance (e.g. York), minimal amounts (e.g. Arrington) or absent (e.g. 'Lady of the Sarcophagus', Milan) while textile preservation ranged from exceptional (e.g. Trier), to small fragments (e.g. Alington Avenue, Dorchester), to mineralised (e.g. Eagle Hotel, Winchester) or no longer extant (i.e. impressions on plaster, e.g. Poundbury). So, beyond the obvious fact that the more substantial (i.e. stone and/or lead-lined) and, importantly, the more intact the container, the better protected its contents (and vice versa, see **7.9**), there does not seem to be a proxy for determining the survival of chemical evidence denoting the treatment of the body with aromatic exudates.

8.5.2 Anthropogenic action

Similarly, the issue of ancient anthropogenic modification may have impacted on chemical composition and degradation. Previous research has shown that resins and gum-resins were frequently mixed with other substances and may have been heated in order to facilitate application to the body (e.g. as part of Egyptian mortuary practices, in the manufacture of Roman ointments and medicaments) or textile wrappings (indications of pasted layers from Trier) (c.f. Colombini *et al.* 2000, 2009; Reifarth 2013: 91-103; Serpico 1996: 358-392; Stacey 2011). These observations receive some support from the primary sources (**Chapter 3**) as Roman authors refer to the use of resinous exudates as part of complex mixtures employed in the creation of perfumed unguents which may have been used to anoint the dead (e.g. *Pliny NH 13.1:3* nd; *Lucian On Funerals 11-15* nd). The ritual use of resins, frankincense in particular, as incense (e.g. *Pliny NH 12.30-32* nd) is also widely attested. It is possible, therefore, that the remaining 'ashes' were scattered over the body in its final resting place as it appears that it was taboo for materials associated with the dead to cross back into the sphere of the living (Erker 2011). Certainly, combustion on the pyre of significant quantities of aromatic substances, when cremation was employed as the method of disposal, is evident from these accounts (e.g. Hope 2007: 111). In inhumation burials, however, it is unclear if these materials were burnt at the graveside or deposited unburnt with the body. Likewise, unburnt offerings including incense (with this term probably used to denote resinous substances in general) might be added to the urn/grave after the remains had been gathered from the pyre (e.g. *Persius Satires 6:34-36* nd).

In the archaeological record, distinguishing between the deliberate pyrolysis of resinous substances and natural environmental degradation presents a considerable challenge. This is especially true if the temperature of the burn was low or highly variable as seems likely given the evidence from Roman period cremation burials (McKinley 1994, 2014) and the fact that, when resins/gum-resins are burnt as incense, they are not fully combusted (personal experience). Indeed, the analysis of samples collected from medieval incense burners has revealed similar findings (Baeten *et al.* 2014). The problem, when considering traces of these archaeological materials, is that the degradation pathways of the precursor resin acids follow similar trajectories regardless of whether the changes are natural or induced. Thus, a pattern of dehydrogenation, decarboxylation and rearrangements resulting in increased aromatization arises, primarily as a result of oxidation (c.f. Egenberg *et al.* 2002). In addition, methylation can occur in various ways due to the release of methanol during the combustion of resinous woods (Hjulström *et al.* 2006), the inclusion of charcoal to promote the burning of incense and, perhaps, even through interactions with degraded woody plant matter within the burial environment (e.g. bier, stems of floral tributes).

In certain circumstances, the appearance of the residue may help to differentiate between these options. For example, there can be no question of heating in relation to the substantial 'lumps' of *Pistacia* spp. resin deposited with the 'Lady of the Sarcophagus', Milan (Bruni & Guglielmi 2005). This is not necessarily a guide, however, since many resins are extruded or extracted as viscous liquids and may remain in this state provided they are kept in sealed containers. Those with a considerable essential oil component such as Pinaceae and balsamic resins stay liquid for extended periods as evidenced by the *L. orientalis* extracts obtained for this project which maintained their fluidity for three years with no visible alteration. Indeed, even a *P. lentiscus* sample freshly collected from Greece in 2011 and stored in a glass vessel remained largely viscous throughout the course of the project although some crystallization occurred after exposure to air for the removal of sub-samples. Thus, although the residues from Grave 8, Poundbury, Dorchester (**7.8**) comprised dark smears on the plaster, the array of resin acids with few derivatives in the largest may denote a naturally degraded Pinaceae resin rather than a tar or pitch (i.e. extensively heated). Other exudates, such as the gum-resins frankincense and myrrh, harden more readily and were probably transported as solid fragments. This is indicated by Pliny's description of the sorting of different grades of frankincense in Alexandria under draconian conditions: "*the workshops can never be guarded with sufficient care; a seal is even placed upon the workmen's aprons, and a mask put upon the head, or else a net with very close meshes, while the people are stripped naked before they are allowed to leave work*" (*NH 12.32:59-60* nd).

8.5.3 Condition 1: a matter of mixtures

Consideration of these factors, in relation to the finds from Roman Britain, reveals that a number of mixtures were identified. Terpenic compounds representative of more than one resinous substance were present in six burials (#5 inhumations including G530, Poundbury and the Mersea Island barrow cremation; **Table 8.1**). Each of these contained a contribution from a triterpenoid (*Pistacia* spp., *Boswellia* spp. and/or *L. orientalis*) and diterpenoid (Pinaceae) source with the latter comprising the minor component. This pattern seems too common to be random, both in terms of the results from this study and previous chemical evidence from Roman period inhumations in continental Europe (e.g. Devièse 2008: 115-131; Reifarth 2013: 91-114; **Table 3.4**). It may, therefore, indicate a deliberate, symbolic, act whose significance escapes us. This could, further, be reflected in the distribution of the *Pistacia* spp. and Pinaceae resins accompanying the 'Spitalfields Lady', London whose relative abundances suggest that they were focussed in different regions of the body (head/torso and pelvis, respectively, **7.4**). The selective positioning of resins may, similarly, be detected in the location of the finds with the 'Lady of the Sarcophagus', Milan (*Pistacia* spp. either side of the head and between the legs; Bruni & Guglielmi 2005) and the recovery of fragments of *Pistacia* spp. resin from around the cranium of the Arrington infant (**7.2**).

It is also possible, in some cases at least, that the Pinaceae resin was present only as an adulterant since the ‘cutting’ of more expensive resins with conifer products is attested in the Roman period (*Dioscorides 1:81* nd; *Pliny NH 12.32:65* nd): *“It [the pitch tree] gives out considerable quantities of resin, which is...so similar in appearance to frankincense, that when mixed, it is impossible to distinguish them; hence the adulterations we find practised in the Seplasia* [an area of perfumer’s shops in Capua, Italy] (*Pliny NH 16.18:40-41* nd). The low abundance of Pinaceae resin markers in relation to frankincense in the Mersea Island barrow cremation could, therefore, be an example of this unscrupulous ancient (and modern) practice. Similar diterpenoid and triterpenoid combinations have been revealed in other archaeological contexts. For example, seemingly deliberate mixtures have been found in an Ancient Egyptian unguent (Pinaceae, *Boswellia* spp.; Mathe *et al.* 2004a), Egyptian mummy balms (Pinaceae, *Pistacia* spp.; c.f. Brettell *et al.* 2015c; Buckley & Evershed 2001, 2004; Łucejko *et al.* 2012), a cosmetic ointment from an Etruscan tomb (Pinaceae, *Pistacia* spp.; Colombini *et al.* 2009) and medieval incense burners (Pinaceae, Cupressaceae, *Boswellia* spp.; Baeten *et al.* 2014). More accidental associations, comprise Pinaceae and *Boswellia* spp. fragments from a house floor in Nubia (Evershed *et al.* 1997a) and what is possibly copal (*Hymenaea* spp.) with frankincense in deposits from a medieval port, Yemen (Regert *et al.* 2008).

The presence of more than one resinous exudate in a number of the Roman mortuary contexts evaluated also implies that other substances are being missed since the compounds detected are, generally, only the more resistant HMM components. The abundance of a transformed animal fat or plant oil (probably the latter as phytosterols and epicuticular leaf wax esters were observed) combined with traces of frankincense and, perhaps, another triterpenic exudate, in the cremation burial (Grave 2) from Purton, Wiltshire, supports this contention as it implies the deposition of a scented unguent. Thus, although the compounds detected in the samples from G336, Winchester (**7.5**), may reflect those present in a Pinaceae and balsamic resin, without secure identification of the latter there can be no certainty that the phenolics and triterpenoids derive from the same source. Likewise, the range of terpenic species (mono- to triterpenes) in the Mersea Island barrow cremation could stem from the Pinaceae and *Boswellia* spp. exudates identified or denote a more complex mixture including essential oil(s). The assortment of sugars, markers of oils/fats and waxes (possibly including beeswax), LMM phenolics and HMM di- and triterpenic compounds in the exceptionally well-preserved sarcophagus burials from Trier indicate the potential range of substances absent from British contexts (Reifarth 2013: 96-99). As predicted (**5.6**), therefore, it seems that much of the evidence (more readily degraded or volatile fractions) for perfumed unguents or deposits composed of a variety of aromatic plant materials (as described in the primary texts, **3.2**), has been lost as the result of natural taphonomic processes.

8.5.4 Condition 2: burning issues

When considering the question of burning or heating, the majority of the finds from Britain (and those from continental Europe as far as can be ascertained from the literature) can be considered consistent with natural, if sometimes fairly extensive, aging as a result of environmental degradation with only ambiguous indications for the changes that might be wrought by pyrolysis. For example, the *Pistacia* spp. fragments recovered from the lead-lined coffin of the infant interred near Arrington, Cambridgeshire were still dominated by their characteristic resin acids. Compounds often lost over archaeological time (e.g. tirucallol) were also present and proposed markers of thermal alteration were either absent (compounds with base peaks at *m/z* 453) or in low abundance (28-norolean-17-en-3-one). Likewise, where a combination of *Pistacia* spp. and Pinaceae resins was identified (‘Spitalfields Lady’, London; Grave 1, Purton) an array of precursor acids and early stage derivatives were present in both with some more degraded species (ocotillones, didehydroabietic acid, 7-oxo-dehydroabietic acid) in the samples from Purton. Defunctionalised compounds symptomatic of more extensive alteration and/or thermal processing were only observed in trace amounts (e.g. 28-norolean-17-en-3-one) or not at all (e.g. retene, methyl dehydroabietate).

These resins had certainly not been burnt as incense and it seems most likely that they had been deposited in their natural state (i.e. unheated), with those from Grave 1, Purton somewhat more degraded than the particularly well-protected samples from the intact sarcophagus of the 'Spitalfields Lady', London (although this could also be due to the longer post-excavation history of the former). The range of compounds in the grave deposits from YORYM:2010.1219 (a plaster burial from York which was discovered in 1877 and lifted in its entirety) may, likewise, support the presence of a degraded but unheated *Pistacia* spp. resin. Despite the near absence of the resin acids, with only a trace of oleanonic acid present, its early stage derivative oleanonic aldehyde was dominant. This was accompanied by 28-norolean-12-en-3-one, its re-arrangement product 28-norolean-17-en-3-one, and a number of ocotillones. Similar combinations have been observed in modern *Pistacia* spp. exudates that have been curated for extended periods of time but whose history precludes deliberate heating. Indeed, as noted above (**8.4**), the diagnostic resin acids (especially the tirucallanes) have been found to be absent or in low abundance in some of these reference resins (**Appendix 3.2**).

With respect to the Pinaceae products, trace amounts in four of the burials from Poundbury (Graves 127, 517, 529, 530) could be the result of either long-term post-excavation exposure to oxidation or thermal processing in antiquity. These residues contained only defunctionalised compounds including retene and/or methyl dehydroabietate (MDHA) which were, however, also present in samples from three other plaster body-casings from Poundbury (Graves 8, 892, 1040) where functionalised compounds (DHA; 7ODHA) remained. Moreover, the most extensive residue from Grave 8 contained a range of resin acids (pimaric, sandaracopimaric, *iso*pimaric and abietic acids) alongside these derivatives. Thus, although potentially the result of variable heating during the production of a tar, the thin layers sampled (pimarane acids only tend to survive in thick masses of tar), presence of low levels of retene and MDHA in modern, unheated, Pinaceae exudates and trace amounts of these compounds in the archaeological samples suggests otherwise. These end products tend to be in far higher abundance in tars (and pitches) which, additionally, contain phenanthrenes and are dominated by DHA and abietic acid (c.f. Egenberg *et al.* 2003; Hjulström *et al.* 2006) as observed in the reference tar (**Appendix 3.1**).

This view is supported by the results obtained from the analysis of materials from the focal burial (G336) at the Eagle Hotel site, Winchester where traces of MDHA were observed in the majority of the samples from within the lead-liner but were accompanied by resin acids (pimaric, sandaracopimaric acid) and DHA in the two (WN12; WN13) that did not appear to have been processed after excavation. The latter also provided evidence of a balsamic resin which had almost certainly not been subject to any degree of heating due to the dominance of the resin acids, absence of any derivatives and survival of LMM phenolic compounds. Thus, it seems that MDHA should simply be viewed as the most persistent indicator for the inclusion of a Pinaceae resin in this burial (and perhaps elsewhere) rather than as evidence of thermal alteration prior to deposition.

The situation with regards *Boswellia* spp. resins is even more difficult to determine as, although pyrolysis experiments have provided a considerable body of data (e.g. Başar 2005: 151-184; Mathe *et al.* 2007; van Bergen *et al.* 1997), far less information is available about the impact of natural degradation. In the samples from Alington Avenue, for example, defunctionalised 24-nordienes were dominant, the boswellic acids were only present in trace amounts and their *O*-acetyl derivatives were absent but so were 24-nortrienes which are considered to be markers of heating (c.f. Başar 2005: 151-157; Mathe *et al.* 2007; **Table 5.6**). This pattern contrasts with the abundance of functionalised compounds present in modern *Boswellia* spp. gum-resins (**Appendix 3.3**) and mirrors their reduction and the concomitant increase in neutral derivatives produced by pyrolysis (**Figure 8.1**). Nonetheless, re-analysis of the reference resins after careful curation (in glass vials in a cupboard for most of the time) for only nine months produced near identical changes to those reported as a result of combustion (**7.11.3.3**; **Figure 7.78**). Thus, these modern materials displayed a dramatic reduction in the boswellic acids and their O-acetyl derivatives, an high abundance of

24-nordienes and traces of proposed markers of thermal alteration (e.g. 24-nortrienes and 24-norursa-3,12-dien-11-one). It is clear, therefore, that much more research is required in order to fully address this question.

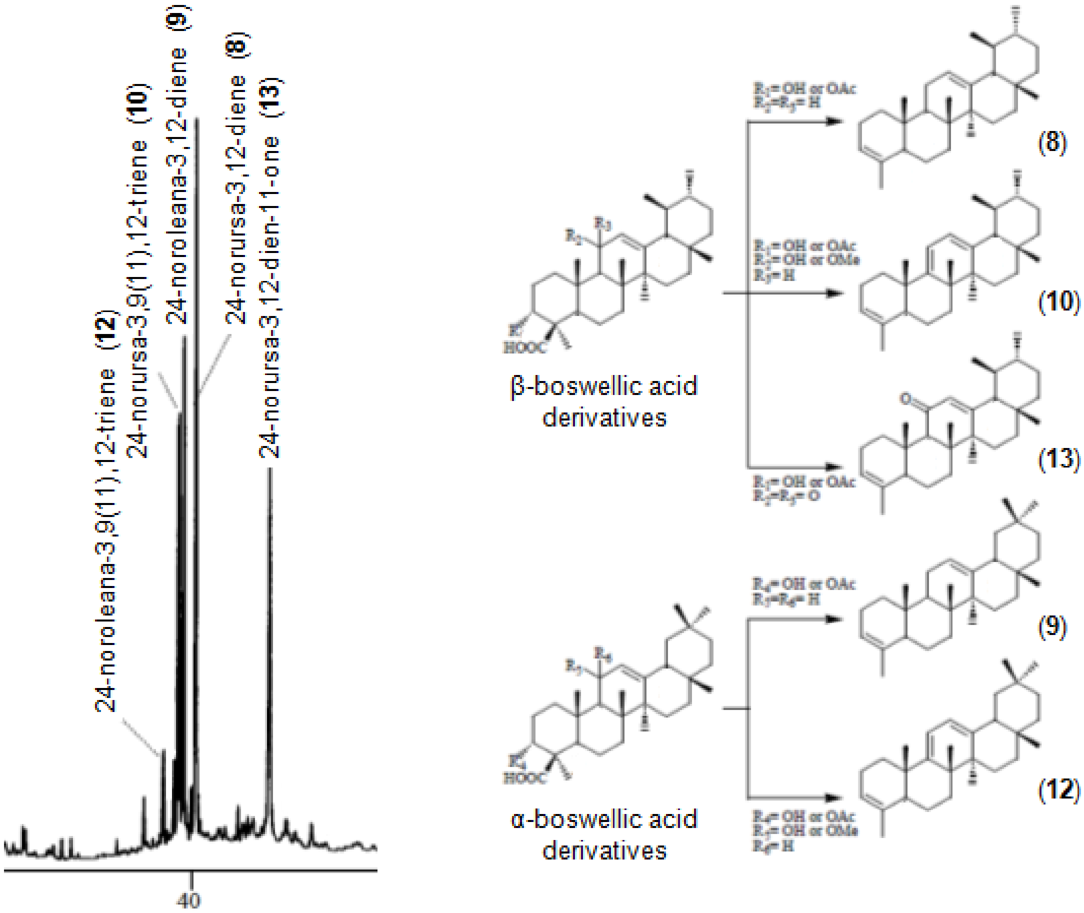


Figure 8.1. Triterpenic fraction produced by the pyrolysis of *Boswellia carterii*: a. partial total ion current chromatogram (30-50 min) (Figure modified from Başar 2005: Figure 5.016, 155); b. schematic showing the nortriterpenes detected and their relationship with their *Boswellic* acid precursors (Figure from Başar 2005: Figure 5.107, 156).

As a result, no conclusions about pre-treatment can be drawn with regards to the two plaster burials from York (YORYM:2007.6205i; YORYM:2007.6206), as the presence of frankincense is denoted only by a combination of 24-nordienes, diagnostic derivatives of the boswellic acids, and the degradation resistant triterpenic alcohols, β- and α-amyrin, their epimers and oxidation products. Likewise, the similar array of triterpenic compounds observed in Grave 2 from Purton could represent traces of frankincense which had been cremated on the pyre. Their association with a transformed fatty residue, possibly a plant oil (found floating in a watery liquid and subsequently curated in that manner) and the observations arising from the degradation experiment

(**7.11.3.3**), however, suggests that natural degradation of a scented unguent provides a more parsimonious explanation. Moreover, in the case of the Mersea Island barrow cremation there can be no doubt that the frankincense was deposited as an unburnt offering within the urn. This is due to the survival of a range of mono- and sesquiterpenes (lost at temperatures in excess of 50 °C; Hamm *et al.* 2003) alongside the combination of cembrene-based diterpenes and triterpenic components unique to *Boswellia* spp. gum-resins and diterpenoids characteristic of Pinaceae resins. Even here, despite the considerable quantity of frankincense recovered, only traces of the boswellic acids were observed with the chromatograms dominated by 24-nordienes. This, again, may be due to changes wrought by immersion in a liquid (probably long term during interment in the barrow) and/or exposure to light and air (the glass vessel and its contents were, reportedly, on display for 100 years in a museum cabinet, Howlett 2013: 15).

8.5.5 Protection and preservation

The fact that this evidence remains at all attests to arrested decay as a result of the additional protection afforded by the use of more substantial, often multiple, burial containers (glass, lead, stone). These findings suggest that preservation was facilitated by a high moisture and low oxygen environment with survival of the precursor resin acids due to retarded oxidation as evidenced by the results from intact sarcophagi and/or lead-lined coffins (e.g. ‘Spitalfields Lady’, London; Arrington infant; Grave 1, Purton). Such anaerobic/anoxic conditions might also account for the general absence or low abundance of acetyl (CH_3-C(=O)-) derivatives in the archaeological samples in contrast to their high abundance in modern, recently solidified, exudates. Transformation of the residue, probably human body tissue, adhering to the hyoid bone in Grave 1, Purton into adipocere appears to support this interpretation. That transformed body fat can be retained over extended periods as a result of fluctuating groundwater conditions is attested by the recovery of a significant quantity of adipocere from a late Roman sarcophagus burial of a child from Mainz, Germany (although the presence of other organic traces were not assessed, Fiedler *et al.* 2009).

Likewise, preservation of the amorphous residues from Grave 2, Purton appears to be due to the waterlogged conditions within the glass vessel (evident during excavation) which had a similar impact on what may be a scented plant-oil. Even in these more protected environments, however, it was noted that the diagnostic precursor acids in the *Boswellia* spp. gum-resins appeared to be more extensively affected (absent or in low abundance) than those in the Pinaceae and *Pistacia* spp. resins (assuming the frankincense had not been burnt as incense prior to deposition). This defunctionalisation, which resulted in an abundance of 24-nordienes, in contrast to the acetylation (presumably produced by initial exposure to air) observed in newly purchased/exposed exudates (with little change in the pentacyclic alcohols and related compounds) was also noted in the modern reference samples after curation (**7.11.3.3**). A similar pattern was observed in the unburnt offering recovered from the Mersea Island barrow cremation vessel which was, again, part-filled with water on discovery. Moreover, as many of the more resistant compounds in frankincense are of relatively common occurrence (e.g. the triterpenols), these factors might account for its infrequent identification in archaeological record.

The method by which LMM components were retained is also a matter of interest. The observations made above, suggest that the mono- and sesquiterpenes in the Mersea Island find may have survived, not only due to the abundance of frankincense (and accompanying exudate(s)) deposited, but because inner regions were protected from evaporation (even during display). Thus, resinous orange fragments appear to have survived by becoming embedded within the white amorphous mucilage produced by interactions with the surrounding liquid (as demonstrated experimentally, **7.11.3.3**). It is interesting to speculate on the possibility that this process would have been facilitated by the deposition of a liquid as a libation (e.g. wine or water) alongside the resinous offerings although, given the island location of the barrow, subsequent water ingress is also highly likely.

The apparent survival of phenolics in the primary burial (G336) from the Eagle Hotel site, Winchester is even more remarkable. This evidence was extracted from mineral-replaced textiles and re-deposited lead, so it seems that adsorption onto these materials (which had themselves undergone taphonomic change) had occurred. It is certainly evident that, where lead coffins or liners were employed, metal ion substitution into plaster body-casings, the mineralisation of textiles and the survival of hair frequently occurred (e.g. Arrington, Poundbury, York). Indeed, the antimicrobial properties of lead were knowingly employed by the Romans to help preserve scented unguents and other organic matter (*Pliny NH 13.3, 34.49-54* nd) which must, surely, be significant in relation to its use in these mortuary contexts. Thus, in conjunction with the hypothesis that epimerisation of the triterpenoids in G336 was catalysed by metal ions this link with unusual preservation conditions appears to make sense. The effect of such conditions on other terpenic compounds is uncertain although a similar impact might be expected where other epimers (stereoisomers, as perhaps observed with the triterpenols in this burial, **7.5.4**) were present with transformation of structural isomers (e.g. pimaric/*iso*pimaric acid; moronic/oleanonic acid) less likely.

8.5.6 Purpose: practical considerations

On balance, therefore, it appears that either a single resin or a combination of at least two resinous exudates containing HMM di- and/or triterpenic compounds were employed in these burials (although these could have been fixatives in unguents which included other plant products composed of LMM compounds) and that, in most cases, they had not been heated (i.e. burnt as incense or extensively treated to produce tars or pitches) prior to deposition. Indeed, the use of scented substances in this manner makes perfect sense in terms of their chemistry since only then would the release of their more volatile components have been able to mask the odour of decay (c.f. Reifarth 2013: 136). In addition, these aromas would have acted to deter invasion of the corpse by necrophagous species (e.g. blow flies, coffin flies, mites), as would tightly wrapping the body in textiles (Forbes *et al.* 2005), while the anti-microbial properties of the resin fraction would have impeded the actions of decomposer organisms. The longer the body was required to be displayed for social or ritual reasons, the more pressing the need for such practical considerations.

This argument is supported by indications that the resinous substances were generally located in close proximity to the human remains. For example, in the lead-lined coffin burial from Arrington, the solid fragments of *Pistacia* spp. resin were recovered from the mass of material including hair and textiles surrounding the head of the infant (Taylor 1993; **7.2**). In Grave 1, Purton (**7.7**) and the child's burial, G4378, Alington Avenue (**7.8**) traces of *Pistacia* spp. and *Boswellia* spp. resins, respectively, were present in residues on the skeletal elements as well as being dispersed in associated debris and grave deposits. At Poundbury (**7.8**) and York (**7.10**), Pinaceae and *Boswellia* spp. exudates again appear to have been applied to body or the textile wrappings since they had come in contact with the inner surface of the plaster with evidence of *Pistacia* spp. resins obtained from grave deposits associated with *in situ* hand bones within the sarcophagus of the plaster burial YORYM:2010.1219, York.

It would also appear from this contextual evidence that, in the inhumation burials, resinous substances were employed in the treatment of the body prior to deposition in the grave rather than scattered over the remains as offerings. The absence of terpenic compounds in the samples from above the lid of the lead-liner, Eagle Hotel, Winchester, the outer surface of the plaster body-casing, Grave 8, Poundbury and the majority of those between the lead-liner and sarcophagus of the 'Spitalfields Lady', London provides further confirmation. Likewise, data obtained from the lead inner coffin of the 'Spitalfields Lady' where spatially-distributed samples were able to be analysed (**7.4**) corroborates this view. Here, the mixture of unheated *Pistacia* spp. and Pinaceae resins were found from head to toe but in far greater abundance in the midline with only minimal traces in the samples beyond what would have been the contours of the body (between the arms and sides of the coffin). This strongly indicates an intimate association with the human remains. The incorporation of layers of resinous substances within the purple-dyed and gold-decorated silks and other textiles used to dress the individuals interred in sarcophagi, within vaults in Trier (Reifarth 2013: 185-430) can, therefore, provide some impression as to the original sensory impact of these beautiful and fragrant dead from Roman Britain.

Precisely how these resinous substances were applied, however, remains unresolved with microscopic evidence from the Trier burials suggesting that a variety of different methods may have been used (i.e. liquids of varying viscosity, possibly containing crushed resins, were coated onto the textiles prior to wrapping the body or poured over the head and upper torso of the enshrouded individual, Reifarth 2013: 91-94). Presumably the method of application would have been affected by the form in which these exudates were able to be transported. As noted above, some (e.g. frankincense) rapidly solidify into 'tears' and appear to have been traded as solids (*Pliny NH 12.32:59-60* nd). These may have been crushed and layered within the textiles or placed around the body with adjectives such as heaped and piled repeatedly used by classical authors (**Appendix 1**). Others, such as *Pistacia* spp. and Pinaceae resins, appear to have remained as viscous liquids. This is evidenced by finds from *amphorae* used as transport vessels and the use of *Pistacia* spp., in particular, as a varnish in Egyptian mortuary contexts (Serpico 1996: 290-357, 405-434; Serpico and White 2001).

Moreover, techniques for promoting the malleability of resinous exudates clearly existed in antiquity as indicated by the creation of effigies from frankincense and cinnamon (*Plutarch Lives Sulla 38:2* nd) and numerous references to perfumed oils and balms which might be smeared on the corpse or used to drench the hair (**Appendix 1**). The discovery of Etruscan and Roman period ointments/medicaments, some of which contained terpenic compounds demonstrates their skill (Colombini *et al.* 2009; Evershed *et al.* 2004; Stacey 2011). Thus, the use of fresh resins (as specified for use as varnishes in Ancient Egyptian texts, Serpico 1996: 342) or manufactured aromatic products would have permitted these scented substances to be spread over (i.e. as part of an cream-like unguent), pasted onto (i.e. in more fluid form, possibly dispersed in a vegetable/leaf oil) or impregnated within (perhaps by pre-soaking) the textiles.

The recovery of evidence for resinous exudates (*Boswellia* spp. mixed with Pinaceae, *Pistacia* spp.? and essential oils) in the two cremation burials provides additional insights into aspects of this rite. In Grave 2, Purton, although it is possible that these scented substances that had been cremated with the body, their addition to the urn prior to final disposal as part of a scented oil or unguent appears more likely due to their association with an abundance of fatty matter, possibly a plant oil. Certainly, the large quantity (over 90 g) of resinous material in the cremation vessel at the centre of the Mersea Island barrow denotes an unburnt offering, presumably deposited in solid form. Thus, the latter, and perhaps both of these finds, appear to have served no practical purpose which indicates that the deposition of scented substances also had ritual significance within the mortuary sphere. Indeed, the presence of clear liquids in both of these burials may indicate that libations (e.g. wine, water) had been poured over the collected remains (c.f. Gee 2008; Jensen 2008) although no markers for wine were detected.

The chemical data obtained from this systematic study of organic residues from Roman mortuary contexts within a single province has, therefore, provided insights into the survival of evidence for resinous plant exudates. It has also begun to reveal practical, and hint at ritual (explored further in **8.7**), aspects related to their use. But how do these findings relate to the broader burial population of *Britannia* and practices across the Roman world?

8.6 Positioned in the world: internal and external relations

As discussed in **Chapter 4**, in Britain the arrival of Rome resulted in an accelerated trajectory towards the disposal of the dead within delineated burial grounds. The final act of separation, in contrast to the archaeologically invisible practices and disarticulated remains characteristic of the PRIA, became marked by containment. Thus, the normative rite, adopted across much of the south and east in the initial post-conquest period, consisted of cremation followed by deposition in a ceramic vessel or other container. Over time, this disposal method was largely replaced, throughout the province, by supine, extended inhumation in a wooden coffin, reflecting changes elsewhere in the Empire. Consequently, there can be no doubt that deposition of the remains of the deceased in substantial, often multiple containers, whether they had been cremated (barrows, tiles, lead, glass/ceramic vessels) or inhumed (mausolea, stone, lead coffins) formed a distinctive, introduced rite. That subsets of the latter practice also involved additional ways of enclosing the body (plaster body-casings, shrouds) and/or the inclusion of resinous substances has now been established. So, how do these material aspects relate to one-another and to normative mortuary rites in Britain and continental Europe?

8.6.1 The material evidence

Of the more than 4500 Roman period inhumation burials recorded from the urban areas and rural burial grounds from which materials for this project were sampled, only around 200 (c. 4%) had been interred in stone and/or lead lined coffins. Samples were obtained from thirty nine, including the solvent wash of the hair from Crown Buildings, Dorchester. A number of these may have been placed within mausolea (e.g. Poundbury) although difficulties in identifying above-ground structures due to centuries of subsequent occupation (e.g. London, York) does not permit any reliable conclusions to be drawn in this respect. Nonetheless, these findings suggest that only a small percentage of the population were provided with more substantial containers. When they were, it seems that a considerable proportion were accorded elaborate body treatments as analysis provided positive results, demonstrating the presence of resins and gum-resins, from sixteen (41%) of those assessed (and two cremation burials). This is a significant percentage especially given the range of taphonomic factors impacting on the survival of such evidence (**8.2, 8.5**).

That these individuals were perceived as different from the norm appears to be supported by the presence of fragments of fine textiles, including wool and damask silk, with high quality decorative elements, gold threads and dye-stuffs such as murex (‘Tyrian') purple, in five cases (e.g. ‘Spitalfields Lady’, child from Alington Avenue). Mineralised textiles or textile impressions were evident in the majority of the remainder (#9). In addition, eight of those interred with resinous substances (#5 from Poundbury, #3 from York) had been provided with extensive plaster body-casings (although #12 had been reported in the literature as plaster burials, for example, the finds from

Alington Avenue, Dorchester which, upon analysis, proved not to be plaster burials; Schotsmans 2013: 195; **7.8**). The rest either showed no sign of white materials (#2) or contained traces (#6) which could have derived from a number of sources including ingress from the surrounding geology and/or the degradation of lead-liners.

This is not only an issue in terms of the misidentification of plaster burials due to the water-solubility of calcites and potential for inwash, but because outwash could have dispersed any plaster present potentially carrying with it other evidence of body treatment (e.g. textile impressions, resinous traces). That garments and textile wrappings, perhaps of high quality, were more widely employed seems certain. Indeed, mineralised fragments and impressions were observed in many of the more substantial containers (e.g. from Poundbury) where resinous substances were not identified. In addition, twenty-three of the stone sarcophagi/lead-lined coffins where secure indications for the use of plaster, including extensive body-casings, were evident and none of those described as plaster burials but interred in wooden coffins were found to have been treated with plant exudates. Despite the difficulties arising from differential degradation and sample type/availability, these observations seem to indicate a correlation between the use of resinous substances and interment with fine, decorated textiles in substantial containers but no clear relationship with the application of plaster.

The presence/absence and nature of any extant grave goods (i.e. ceramics, metal wares, jewellery) also shows no clear patterning although those accompanied by the best quality material possessions were found to contain resinous substances but were not, generally, plaster burials (e.g. 'Spitalfields Lady', London; Alington Avenue child; possibly Grave 1, Purton). A chronological argument could be put forward, here, in relation to the general decline in the deposition of grave goods and increase in the use of plaster as part of burial practices throughout the 4th century AD. Thus, the unaccompanied burials (e.g. Poundbury, late 4th century AD) are probably later in date than the accompanied (Alington Avenue, 3rd century AD; Purton, early 4th century AD; 'Spitalfields Lady', mid 4th century AD). This is pretty speculative, since the Arrington infant (2^{nd}-3^{rd} century AD, pipeclay figurines, possibly plaster) and the focal burial at the Eagle Hotel site (early 4^{th} century AD, coin only, no plaster) do not seem to fit this pattern, while no clear details are available with regards those from York.

In addition, the cremation burials evaluated (Mersea Island, 2^{nd} century AD; Grave 2, Purton, date undetermined) were not accompanied by unburnt grave goods (some cremated animal and bird bones were recovered from the latter). The identification of resinous substances in these glass vessels (associated with a quantity of clear liquid in both and an abundance of fatty matter, in Grave 2, Purton), however, supports suggestions made in the 1800s for the presence of exotic offerings with other cremation burials in Britain. Thus, gums, gum-resins, honey and plant oils may have been deposited below the barrows at Bartlow Hills, Cambridgeshire in the 2^{nd} century AD (Gage 1834, 1836) and the residue of a gum-resin have been present on a Samian ware dish accompanying a cremation *amphora*, at Weston Turville, Buckinghamshire (Waugh 1962). In these cases, a notable array of unburnt grave goods were also present (**Figure 8.2**). Thus, the provision of grave goods displays appears highly variable and bears no obvious relationship to the presence of resinous exudates.

Figure 8.2. The extensive range of grave goods found with the cremation burial in a clay *amphora*, Weston Turville, Buckinghamshire, UK (Figure from Isham 1855: 77).

A similar pattern can be observed in the continental finds. Those treated with resinous substances had all been interred in sarcophagi (with the exception of the multiple burials in the catacombs) and clothed in gender-appropriate garments (e.g. tunics, dresses, cloaks). These textiles were, again, made from wool, linen and damask silk often with evidence of purple-dyed *clavi* (stripes) and gold tapestry-work. In addition, bandages, face-covers and shrouds were identified (Mitschke & gen. Schieck 2012; Reifarth 2013: 47-90; Wild 2013). No clear association in terms of the presence/absence of grave goods could be discerned although an abundance of quality items was more commonly observed with those of earlier date (e.g. Grottarossa girl, Rome; 'Lady of the Sarcophagus', Milan; adult and child, Naintré; **3.3**) while the 4th century AD sarcophagus burials from Trier were unaccompanied (except for a pair of shoes in the plaster burial of a child; Reifarth 2013: 147-148).

The disjunction between the inclusion of plant exudates and plaster was, likewise, evident. Thus, some individuals accompanied by resinous substances, textiles and/or grave goods of the highest quality (e.g. jewellery, gold hair nets, glass vessels, items of amber and ivory) were not encased in plaster (e.g. Chioffi 1998: 66-67; Papageorgopoulou *et al.* 2009; Maspero and Rottoli 2005) although others were (Devièse 2008: 145-148). The best evidence as to the range of possibilities encountered comes from St. Maximin's, Trier where textiles incorporated with resinous fragments were associated with individuals interred with plaster, accompanied by aromatic woods (*Abies* spp. or myrtle twigs) or neither while four burials (one plaster, one with wood shavings, two with neither) contained no evidence of terpenic compounds (Reifarth 2013: 185-430).

8.6.2 The human factor

So what can be discerned regarding the individuals who had been treated in this manner? All of those inhumed with resins or gum-resins (for whom data is available) were interred supine, extended which is consistent with the standard body position employed across the Empire in late Roman period. In the British examples, various grave/body orientations are recorded. These generally reflect the predominant alignments within each burial ground (e.g.

west-east, head to the west at Poundbury) with G336, the primary burial at the Eagle Hotel site, Winchester seemingly the only anomaly. This mature male was positioned north-south, head to the north, while the remainder (c. 47 inhumations) at this site were roughly west-east aligned (Teague 2012). Much has been made of the significance of such factors with west-east orientation considered by some a marker of Christian burial rites (c.f. Sparey Green 2003; Watts 1991: 121-128). This link is tenuous and may, in fact, be a matter of chronology as, throughout the 4th century AD, a general trend towards more regularised arrangements (e.g. burials in rows) has been noted. This appears to reflect greater administrative control over the management of burial grounds with the increased standardisation of east-west orientation probably a response to the Imperial adoption of the cult of Sol Invictus in the late 3rd century AD (Esmonde Cleary 1989: 123-130; Philpott 1991: 239-240; Salway 1981: 693-707). That it was also acceptable for followers of Christ to be interred in this way is shown by the epigraphs from Trier (Reifarth 2009).

Evaluation of the osteological and palaeopathological information is more illuminating and has revealed that five males (#1 cremated; #4 inhumed), seven females (#1 cremated; #6 inhumed) and six unknowns (all inhumed) had been interred with resinous substances. These comprised three sub-adults (#1 infant, #1 child, #1 juvenile), one individual aged between 18-25 years (Grave 1, Purton) and eleven adults (>20 years old) whose ages ranged from young (20-25) to mature (>45) for those inhumed (#10) and young to middle adult (25-40 and 35-45 years) for those cremated (#2) alongside three unknowns (**Table 8.1**). Although this sample size is too small to be statistically significant, it is clear that the deposition of resinous exudates as offerings or their use in the treatment of the body was considered appropriate for both males and females and individuals of all ages. Comparison with demographic data from substantial cemetery populations (e.g. Poundbury) or a combination of urban/regional burial grounds (e.g. those around Winchester) shows that the ratio of sub-adults:adults in these ‘resin burials’ falls within previously reported parameters with regards to those inhumed (**Table 8.3**). No conclusion can be drawn from only two cremation burials.

Table 8.3. Age and biological sex distribution in inhumation burials (by number or percentage as given in the reports) from urban and rural areas of Roman Britain. N.B. these represent the skeletal remains that were able to be sexed and aged; it was not possible to sub-divide the age categories as different age ranges were used in each study; *the age range for the young female from Grave 1, Purton is given as 18-25 years (McKinley 1994).

Roman period burial grounds	**Biological sex**			**Age determinations (years)**		
	M/?M	***F/?F***	***Ratio***	***Subadult (<20)***	***Adult (>20)***	***Ratio***
Burials around London Hall 1997	88	55	1.6:1	61	143	0.4:1
Eastern burial ground of London Barber & Bowsher 2000	186	109	1.7:1	25%	75%	0.3:1
Burials around Winchester Ottaway *et al.* 2012: 210-218	59%	41%	1.4:1	38%	62%	0.6:1
Lankhills, Winchester Booth *et al.* 2010	207	214	0.9:1	233	514	0.5:1
Burials from Rural Dorset Redfern 2006, 2008 – adults assessed	56	36	1.6:1	---	92	---
Poundbury, Dorchester Farwell & Molleson 1993	146%	156%	0.9:1	423	766	0.6:1
Burials around Cirencester McWhirr *et al.* 1982	207	93	2.2:1	63	292	0.2:1
Trentholme Drive, York Wenham 1968	231	52	4.4: 1	44	246	0.2:1
Burials around Colchester, Essex Crummy *et al.* 1993	180	146	1.2:1	215	498	0.4:1
Inhumation burials analyzed for this project found to contain resinous substances	4	6	0.7:1	3/4*	10/9*	0.3:1/ 0.4:1

When it comes to the biological sex of these individuals, however, there appears to be a preference in terms of females (**Table 8.3**). This is of particular interest, as an imbalance in favour of a greater percentage of males in burial populations from Roman Britain has previously been noted with a predominance of females rarely observed (Woodward 1993). Indeed, a study of twenty-five urban, Roman period, burial grounds revealed that 62% of those for whom sex could be determined were considered to be male with only 38% ascertained as female, a ratio of 1.6:1 (Davison 2000). Moreover, a general bias towards more males in archaeological burial populations (regardless of time or place) has been widely reported and may be due to osteological sexing techniques (Redfern 2008b). The fact, therefore, that a greater number of apparent females from Britain were accompanied by resinous exudates may be significant since the various osteologists involved in assessing the skeletal remains would all have been subject to the same methodological limitations.

These observations are supported by the continental finds (**Table 3.4**) as resinous substances have been identified in the elaborate inhumation burials of both males (#6) and females (#13) of all ages (e.g. below St Maximin's, Trier those interred ranged from a 3-9 month old infant to an <69 year old adult; Reifarth 2013: 43). Again, a preponderance of females (ratio M:F 0.5:1) is evident in these European burials but also a greater percentage of sub-adults (51%, including two infants in a triple burial). A similar pattern has been noted by Chioffi (1998: 31) in her review of potential examples of embalming and mummification in the Roman period, outside of Egypt. This data correlates with the literary evidence as Roman authors refer to the use of aromatic substances as part of the mortuary rites accorded both sexes and imply that sub-adults as well as adults were treated in this manner (**Appendix 1**). They also indicate that the most lavish materialised manifestations of grief were provided by wealthy elite males mourning their wives or children (Hope 2009: 137-144): *"No words can express my grief...for the money he had intended for clothing...and jewels [for her wedding was now] to be spent on incense, ointment and spices*" (*Pliny Letters 54:16* nd).

Thus, the archaeological record may now be providing evidence of this anguish as the most elaborate combinations of stone, lead, textiles, dye-stuffs and grave goods accompanied by resins and/or gum-resins from Britain were those of young adult females ('Spitalfields Lady', London; Grave 1, Purton) and the child (G4378), perhaps a male, interred at Alington Avenue, Dorchester. The treatment of the year old infant found at Arrington, Cambridgeshire is also notable as this age group appears to have been viewed as liminal (not fully of this world) and so those under approximately 12 months old were generally interred with simple rites, often in domestic contexts (Esmonde Cleary 2000; Moore 2009: 191-196). Likewise, females and children were accorded the most sumptuous treatments among the examples identified from continental Europe (e.g. 'Lady of the Sarcophagus', Milan; Naintré and Anché, France; **Table 3.4**). What we are seeing, therefore, may be one aspect of the emphasis on the family in Roman society manifested through the delineation of female identity which centred around an idealised life course of childhood, marriage and motherhood since such deaths represented a blow to "familial and social continuity" (Moore 2009: 209). The complex treatment of the cremated remains of the individual placed at the centre from the Mersea Island barrow and the substantial grave prepared for the primary burial (G336) at the Eagle Hotel site, Winchester, however, demonstrate that resinous substances were also deemed appropriate for inclusion with mature adult males of significance to their communities.

Consideration of evidence for pathologies affecting these individuals may also indicate something about their social status. Those inhumed with resinous exudates in British contexts showed no evidence of violent injury with only bilateral *cribra orbitalia* (Arrington infant) and *osteitis/periostits* (Grave 892, Poundbury) observed in terms of skeletal manifestations of disease (Appleby 2011; Molleson 1993: 188). Indeed, the majority of those interred in more substantial containers, for whom data could be assessed, provided minimal evidence of trauma or ill-health (e.g. despite the wide variety of injuries and ailments evidenced in the Poundury population only three 'package' burials (G525, G862, G892), all of whom showed traces of infection in the form of periosteal new bone formation, were affected, Molleson 1993: 182-204). In addition, the cremated remains from Mersea Island, Essex displayed non-specific (age, disease or activity-related) excess bone-formation (i.e. osteophytes, enthsophytes, exostoses) on various joints and the lower limbs together with spinal lesions (ossification of the anterior longitudinal ligament and ankylosis) characteristic of diffuse idiopathic skeletal hyperostosis (DISH), probably accompanied by osteoarthritis (McKinley 2014). The precise aetiology of DISH is unclear but it has been linked to obesity and Type 2 diabetes suggesting that this robust, mature adult male may have lived well (Roberts & Manchester 1997: 120-121).

These findings contrast with the widespread evidence for violence and ill-health in the Roman period denoted by traumatic injuries, non-specific markers of environmental/dietary stress and skeletal manifestations of scurvy, rickets and tuberculosis in the broader population and which appear particularly prevalent in urban areas (Bonsall 2013: 313-314; Molleson 1993: 182-204; Redfern 2008b). Even in the western world today, those who are socially and economically disadvantaged, especially during childhood, suffer more severe and/or repeated bouts of illness and have a far shorter life expectancy as a result of poor diet, poor living conditions and less access to treatment (www.who.int/hia/evidence/doh/en). This relationship has been shown to hold true for Roman Britain (Redfern & DeWitte 2011) so, since most of those interred with resinous substances came from burial grounds associated with large public towns, the implications are that they may have been members of the social elite.

These observations are matched by the continental finds where the adult remains show no signs of injury and minimal evidence of disease. Thus, only minor age-related degenerative changes, alongside unremarkable dental wear and tooth loss, had affected the 50-60 year old female from Thessaloniki (Papageorgopoulou *et al.* 2009) while slight lesions, possibly indicative of cancer, were observed on the rib bones of the 24-31 year old 'Lady of the Sarcophagus', Milan (Cattaneo & Porta 2005). In Trier, examples of linear enamel hypoplasia (LEH, evidence of periods of ill-health or dietary insufficiency overcome in childhood) and a single non-specific lesion on the femur of one individual were noted (Reifarth 2013: 43). Some of the children from Trier also showed signs of LEH with Harris lines (growth arrest lines) evident on the femora of the Grottarossa girl from Rome (Ascenzi *et al.* 1996; Reifarth 2013: 43). These, as with the *cribra orbitalia* affecting the Arrington infant, are indicators of episodes of dietary, environmental or disease-related stress and so are, presumably, in line with their early demise although there is no evidence for cause of death.

8.6.3 The place of 'resin burials'

What can be said about the significance of this rite? A simple equation between materiality and identity would be a retrograde step. Nonetheless, it would be churlish not to state that the most elaborate manifestations of this body treatment denoted the social status of the deceased based on the small percentage of individuals apparently interred in this manner, the range and nature of the materials employed, the osteological evidence, the epigraphs from Trier (c.f. Reifarth 2009) and matching classical descriptions of the funerals of members of the Roman elite (**2.3; 3.2**; **Appendix 1**). In the Roman world, the social persona of the living and the dead has been shown to be of paramount importance (c.f. Brooke 2011; Hope 2009: 67-77; **2.5**). This does not mean that inversion did not occur in the mortuary sphere although the form it took would seem to have reflected the hypocrisy of the survivors rather than to have masked the status of the deceased. For example, there is a clear contrast between Nero's behaviour to Poppaea in life (he reportedly kicked her to death in a fit of rage while she was pregnant) and the ostentatious funerary display he accorded her in death (*Pliny NH 12.41* nd; *Suetonius Lives Nero* nd; *Tacitus Annales 16.6* nd). Similar critiques of immoderate displays of mourning, demonstrated through material excess, are found elsewhere in Roman writings (e.g. Hope 2009: 125-149; *Pliny Letters 4.2, 4.7* nd; *The Twelve Tables Table Ten* nd). It should also be stressed that simpler versions of this body treatment, which included the deposition of a few grains in honour of the gods (c.f. *Pliny NH 12.41:83* nd), almost certainly existed but remain undetected (and will probably continue to do so) due to taphonomic factors. Even so, what this combination of archaeological and literary evidence indicates is that the most lavish mortuary treatments were accorded individuals of wealth and social standing (c.f. Philpott 1991: 228-232).

Considering the material aspects of this 'package', the repeated association between resinous exudates and interment in stone sarcophagi and/or lead lined coffins appears to support this contention. These materials had to be mined, transported, shaped, and sometimes decorated, indicating a considerable financial investment. For example, the limestone used in the manufacture of the sarcophagus of the 'Spitalfields Lady', London originated in Barnack, Cambridgeshire (Thomas 1999) while the lead for her decorated inner coffin also came from English sources, possibly Somerset (Montgomery *et al.* 2010). Likewise, the deposition of textiles including silk damask and wool with gold threads and dyestuffs in a number of these burials suggests the conspicuous consumption of costly items symbolic of wealth and status (c.f. Wild 2013). The raw materials used would have come from far afield including China (silk) and the Mediterranean (murex purple) while some of the garments may have been manufactured in the Levant (e.g. tunic with *clavi* worn by the child interred at Alington Avenue) (Walton Rogers 2002).

Of course, as noted above, it is highly likely that taphonomic factors have obscured the more extensive inclusion of plant exudates in Roman period burials (both ‘package’ and less protected, **8.5**). Nonetheless, it appears that they are often associated with the most significant burials from each site or region investigated (e.g. Grave 1, Purton; G336, Eagle Hotel, Winchester; individuals additionally interred in mausolea, Poundbury, Dorchester). As they were not, however, observed in all of the sarcophagus and/or lead-lined coffin burials assessed (with the proviso that sample availability and post-excavation handling (**8.2**) alongside amount deposited and natural degradation will have resulted in many losses (**8.5**)), other factors such as cost and availability must sometimes have restricted access. For example, in the case of the double burial from Boscombe Down, Wiltshire it may be that the rural nature of the region played a part. Indeed, of the around 300 Roman period burials found on Boscombe Down, it was the only one in a stone sarcophagus. This provides a clear contrast with neighbouring Dorchester and its environs and fits with previous research which has noted a correlation between burials with elite characteristics, urban areas and the distribution of wealth in Roman Britain (Struck 2000; Toller 1977: 2). In addition, issues such as personal choice (**8.7**) and the extent of knowledge of this practice (**9.1**) should not be forgotten.

It is also possible that a gum, essential oil (**5.2**), gum-resin such as myrrh (**5.4**) or even a species which, due to natural variability, did not contain the classic biomarkers of its genus (**8.4**) could have been employed in these burials but has left no identifiable chemical traces (**8.5**). That more ephemeral exudates may have been used is indicated by the FTIR results from Trier (Reifarth 2013: 96-99) and palynological data from other continental finds (e.g. Ciuffarella 1998; Ghini *et al.* 2005). This is emphasised in the case of a plaster burial (F77) from Bezannes, near Reims, north-east France (Bontrond & Bouquin 2012). Exotic pollen, possibly of eastern African or southern Arabian origin, was recovered from residues at the base of this decorated lead coffin (Corbineau 2012). Various methods by which these grains could have been transported were considered including incorporation within a resinous substance or unguent. Chemical analysis of various samples revealed evidence of microbially-degraded animal and plant matter with markers for the latter dominated by friedoolean-3-one, a pentacyclic triterpene of widespread occurrence. No clear evidence for the deposition of a resin or gum-resin was recovered (**Appendix 4**). Similarities between these results and those obtained from a residue within a *biberon*, a glass vessel found in a pit (F31) within the burial ground (**Figure 8.3**) are, however, intriguing. The latter was found to contain a mixture of calcium carbonate, hydrolysed animal fat and a degraded plant oil and/or wax (Garnier 2012). Thus, it is possible that an oil or unguent from which LMM volatile aromatic compounds have evaporated had been deposited with F77 although, this cannot be proven.

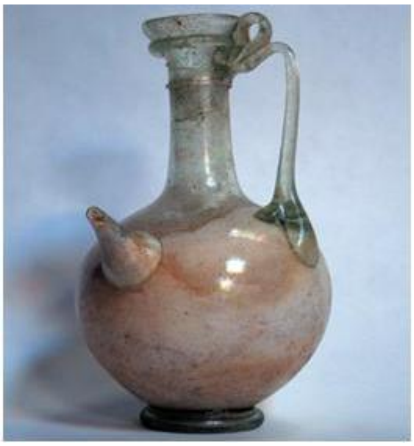


Figure 8.3. Glass vessel (*biberon*) recovered from pit F31, Roman period burial ground, Bezannes, near Reims, France (Image from Garnier 2012: Figure 1, 333).

8.6.4 The role of religion

Establishing a connection between the use of resinous substances and any specific religion or cult is even more problematic. Indeed, given the ritual significance of aromatic exudates across the ancient world, this distinction would probably not have been relevant throughout much of the Roman period (**2.3.1**). Viewed both as gifts from the gods and as offerings appropriate to the gods, in the form of incense they were believed to bear the essence of offerings to the heavens (burning incense, or certain types at least, appears to elicit a psychoactive response, Moussaieff and Mechoulam 2009) while, as part of scented unguents and perfumes employed in anointing rituals, they acted to cleanse and purify the body. Thus, their fragrance was used to propitiate a wide range of pagan deities (Groom 1981: 1-14). That they played a similar role in Jewish and Christian rituals is attested by passages in the Old and New Testaments (e.g. *Bible: SEV* 2009 *Exodus 30:34-35, Leviticus 2:3, 6:15, Matthew 2:1-12, John 19:39*). It was, therefore, only towards the end of the 4th century AD that their use in the mortuary sphere began to be criticised by the early Doctors of the Church (Alcock 1980; Ando 2003; Martorelli 2000).

This universal appeal of resinous plant exudates is supported by their discovery in both cremation and inhumation burials in Britain dated between the 2nd and late 4th centuries AD thereby spanning the pagan and Christian periods. Thus, the Mersea Island cremation (2nd century AD), situated below a barrow and accompanied by an abundant unburnt offering of frankincense, displays links to PRIA/early Roman mortuary practices in eastern Britain and Gaul (**4.2**) and resonates with literary descriptions of rites in Rome around the turn of the first millennium (**2.3; 3.2**). Due to the method of disposal, the cremation (Grave 2) from Purton is also, presumably, pagan regardless of date of deposition. Likewise, the pipeclay figurines interred with the Arrington infant suggest a variety of pagan influences and can be dated to the 2nd-3rd centuries AD on stylistic grounds (Bémont *et al.* 1993, 131; Green 1993; **Figure 8.4**). These mould-made anthropomorphic and animal figures, mass produced in factories in Gaul and the Rhineland, appear to have held considerable ritual significance in the Celtic provinces (Rouvier-Jeanlin 1972). Indeed, most such figurines have been recovered from graves although some seem to be connected with household shrines and others, deposited in sacred springs and sanctuaries, may have been votive offerings or have marked a rite of passage (Rouvier-Jeanlin 1972: 27-29). Other examples from Britain support their pagan aspect as they include a curated

Dea Nutrix (nursing goddess) deposited with another infant (c. 1 year old) at Baldock, Hertfordshire (19 km from Arrington) in the late 3rd-early 4th century AD (Burleigh *et al.* 2006) and two pipeclay *Venus* figurines from the grave of a child interred with high quality grave goods (late 1st-3rd centuries AD) in the eastern burial ground of London (Barber & Bowsher 2000: 186-189, 325). In addition, the primary burial at the Eagle Hotel site (4th century AD) with its anomalous north-south alignment and coin in the hand may suggest pagan practices, although such associations are highly questionable (c.f. Esmonde Cleary 1989: 125; Woodward 1993: 236-237).

Figure 8.4. Pipeclay figurines accompanying the burial of an infant, lead-lined coffin, Arrington, Cambridgeshire, UK (Image from Taylor & Green 1992: 420).

There can, however, be no certainty regarding the religious beliefs of the majority of the individuals from Britain who were accompanied by resinous substances although questions regarding the significance of plaster burials (and, therefore, those containing plant exudates as well) remain. Forensic experiments have shown that all forms of calcitic materials function as temporary preservatives absorbing body fluids and restricting microbial action (Schotsmans *et al.* 2011). Thus, as much of the 'white stuff' from Roman period inhumations has not been analysed while sulfurous compounds released by body degradation might, through adsorption into a lime plaster matrix, mimic gypsum (if surface and core samples are not evaluated), care must be taken when making claims as regards specific types of plaster (c.f. Sparey Green 1977). Seemingly added at the graveside (Barber & Bowsher

2000, 101-103), the application of plaster to the body can be rationalised when sarcophagi were to be placed within vaults or mausolea that would be regularly revisited for acts of remembrance and subsequent burials. What remains inexplicable is its inclusion in otherwise normative soil-cut graves.

Thus, the contention that plaster, regardless of type, held some form of symbolism cannot be simply dismissed and a possible relationship with Christian thought, due to the apparent origins of this rite in Africa and its adoption by the Donatists, provides a plausible argument (Aufderheide 2003: 259; *Herodotus 3:24*; Pettigrew 1834; Ramm 1971; Sparey Green 1977). It is the only material aspect of these substantial late Roman period inhumation burials that has not, as yet, been observed in relation to their earlier cremation counterparts. Turning to the continental evidence, many of those interred in Trier were attested Christians (Reifarth 2009), as those in the imperial catacomb at Rome may also have been (Blanchard *et al.* 2007) while a *chi-rho* was present on one of the lead-liners from Anché, France (Devièse 2008: 64). One avenue to continue to evaluate during future research (**9.2**) is, therefore, whether the various materials employed, all of which acted to temporarily preserve (plaster, wood shavings, textile wrappings, resinous substances) and/or maintain the integrity of the body (textile wrappings, plaster, lead, stone), were adopted by Christians, according to their purse and social standing, due to their correspondence with a developing religious ideology which emphasised physical resurrection.

8.6.5 Origins and influences

What is clear as a result of this study is that the use of resinous substances in mortuary rites extended across the Empire and arrived in Britain by the 2nd century AD, at least, and that their deposition within the grave continued throughout the Roman period. This is important as contemporary authors only provided insights into practices focussed around cremation rites in 1st century BC-2nd century AD, Rome (**Table 3.1**) and previous chemical evidence related, predominantly, to late 3rd-4th century AD inhumation burials. Thus, the finds from the two cremation vessels from Britain form a bridge between archaeology and antiquity and, since commonalties in the materials employed (with the exception of plaster) indicate continuity of underlying concepts regardless of method of disposal, questions about the origins of the use of aromatic resinous substances can be approached.

Examination of the literary evidence indicates that the use of scented substances in the mortuary sphere initially derived, as did so much else in terms of ritual in the Roman world, from Greece (Alcock 1980; Beard *et al.* 1998a: 54-72; Hope 2009: 9-10). From the manner in which death should be faced, through the sequence of preparing the body, to its final disposal (with both cremation and inhumation acceptable) the path followed by the Roman dead mirrored that previously trodden by the Greeks (Garland 2001: 13-37). This is supported by archaeological evidence as, following the extensive colonisation of southern Italy in the 7th-8th centuries BC by migrants from Greece, elements of their mortuary practices have been shown to have spread across the region and beyond (Davies 1977). Thus, in south-central Italy (i.e. Etruria and Samnium) sarcophagus burials are regularly observed, tomb iconography reflects Hellenistic styles and mythological themes and the dead are, generally, interred supine, extended, appropriately clothed and accompanied by grave goods indicative of aspects of the individual's identity (Davies 1977; Toynbee 1996: 14-17).

The social significance of textiles ornamented with Tyrian purple is also attested in the Greek world (e.g. *Homer Iliad 24.1005* nd) with their role in the mortuary sphere demonstrated by the fine purple-dyed fabrics found in a 5th century BC sarcophagus (probably belonging to a member of the family of Alcibiades) in the Keramameikos burial ground, Athens (Margariti & Kinti 2014). That the use of perfumes and unguents, including as part of funerary rituals, derived from contact with Greece is acknowledged by *Pliny* (*NH 13.1-2* nd) with the act of anointing the dead originally associated with the gods (Shelmerdine 1995); "*but the dogs came not about the body of Hector, for...Venus kept them off him...and anointed him with ambrosial oil of roses that his flesh might not be torn*" (*Homer Iliad 23.186-187* nd). Indeed, an alabaster *unguentarium* containing a scented ointment was recovered from a tomb (2nd century BC) in a Hellenistic necropolis (probably belonging to one of the aristocratic family groups) of the Etruscan city Chiusi (Tuscany), Italy and found to comprise a mixture of vegetable oil, *Pistacia* spp. and Pinaceae resins (Colombini *et al.* 2009).

Subsequently, the syncretic nature of Roman religion allowed eastern cults to add their influence as mortuary practices from various provinces reached Rome and then were exported across the Empire. Thus, a small number of examples of mummification in 'true' Egyptian fashion are attested in Italy, for example the Grottarossa girl interred outside Rome (Ascenzi *et al.* 1996; **Figure 3.1**). It must be emphasised, however, that the remainder of these chemically identified Roman 'resin burials' are something quite different, as noted by Chioffi (1998: 24) and Reifarth (2013: 135). This is evident even from visual comparison of this body treatment with that accorded Egyptian mummy bundles (**Figure 8.5a**) and is supported by descriptions of Egyptian mummification practices in the primary sources (e.g. *Herodotus 2.87* nd*; Pliny NH 16.52* nd; Riggs 2010) in conjunction with published evidence regarding the complex mixtures used to create mummy balms (e.g. Buckley & Evershed 2001; Colombini *et al.* 2000; Koller *et al.* 2003; Ménager *et al.* 2014).

As part of this project, the very different nature of the latter approach was confirmed by examination and chemical analysis of samples from nineteen votive non-human mummies and one human mummy (Brettell *et al.* 2005c; **Appendix 5**). Egyptian mummification aimed, relatively successfully, to preserve soft tissue indefinitely. To achieve this end, evisceration was sometimes employed while desiccation with natron was more extensively utilised (Aufderheide 2003: 322-294; David 2000). The application of a complex mixture of organic substances (e.g. plant oils, beeswax, bitumen, conifer extracts, resins) to both the inner cavity and exterior of the body/associated textiles was, however, universal (c.f. Baumann 1960; Serpico 2000). Over time, these balms resulted in an dull, opaque brown-black, often brittle/powdery, coating (due to oxidation) which overlaid and/or impregnated layers of bandages below which skin, muscle and other tissues remained recognisable (Brettell *et al.* 2015c; **Figure 8.5b-c**).

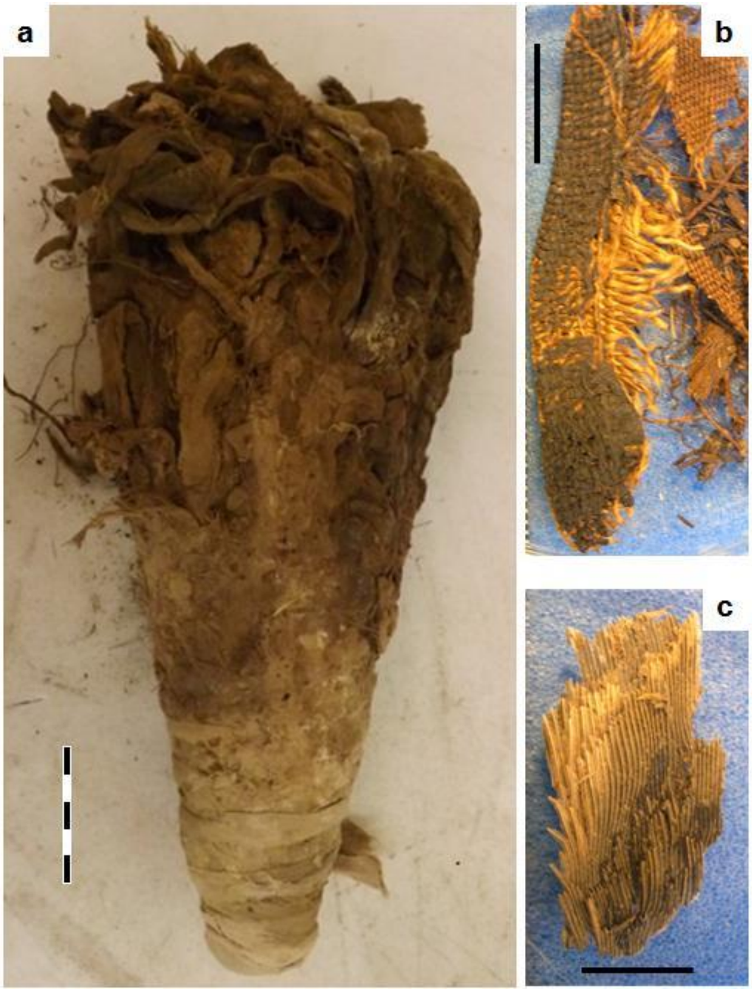


Figure 8.5. The appearance of residues coating Roman period votive mummies: a. ibis bundle, Ancient Egyptian Animal Bio-Bank (AEABB)401 (©AEABB). Scale bar in cm; b. textile fragment, bird of prey, AEABB004; c. wing feather, *Accipiter* sp., AEABB384 (Author). Scale bars = 1 cm.

This was in contrast to the skeletonised remains present in the Roman period inhumations from Britain, even those from Grave 1, Purton where the dark, almost burnt, appearance of the bone was accompanied by extensive brushite crystal formation (based on observations made during this project and by Eline Schotsmans 2012, pers. comm., March-November, such changes relate to the enclosed microenvironments created within many of these lead and stone containers rather than the presence of resinous substances, *per se*). Likewise, the continental evidence, in particular the images from Trier, serve to illustrate the considerable differences in the style and impact of the body treatments employed. Indeed, in these well-protected, undisturbed sarcophagi, conditions were such that even bone was, generally, so poorly preserved that many individuals could not be sexed or aged with confidence (Reifarth 2013: 185-430; **Figure 8.6a-b**).

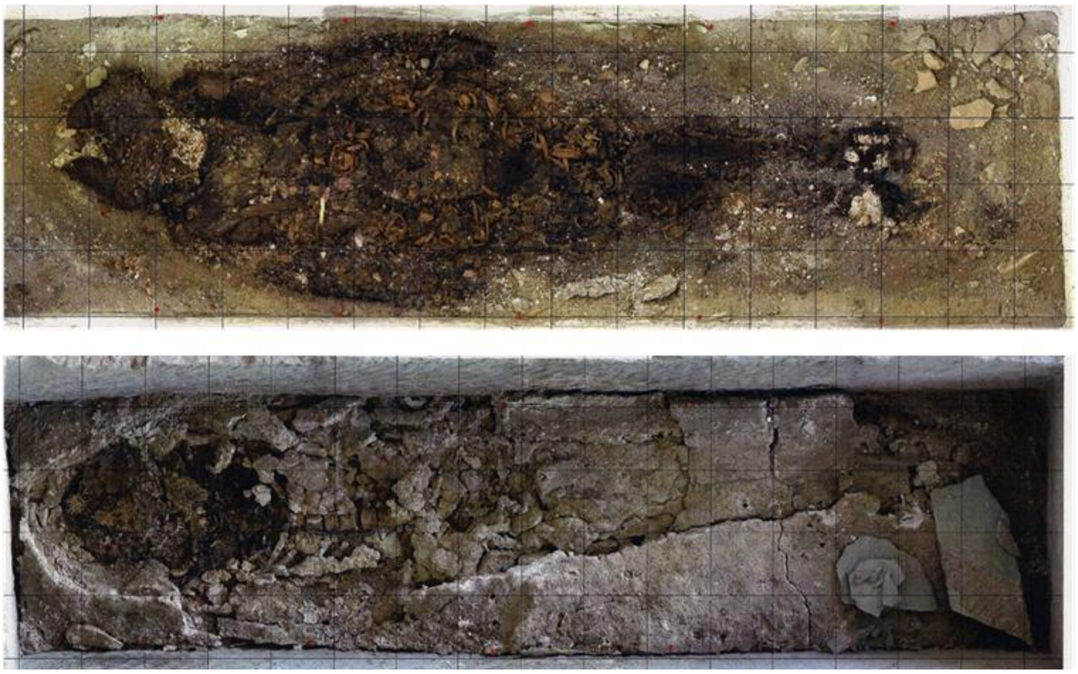


Figure 8.6. Sarcophagus burials from St Maximin's, Trier, Germany: a. Grab 4, interred with *Abies* spp. bark shavings, sub-adult c. 15 years old; b. Grab 107, plaster burial with void over cranium, young adult, female (Images provided by Dr N. Reifarth © Nicole Reifarth).

Chemical data from the dark residues coating the Egyptian mummies, analysed as part of this project, supports this view (Brettell *et al.* 2015c). These materials provided evidence for complex mixtures of fats/oils, plant waxes/beeswax, coniferous essential oils and highly degraded resins (Pinaceae and *Pistacia* spp.). As no precursor acids remained, these materials (alongside the beeswax) appeared to have been quite strongly heated in order to facilitate mixing and application as reported in other studies (c.f. Colombini *et al.* 2000; Jones *et al.* 2014; Serpico 2000). This is clearly at odds with the results from the Roman period inhumations assessed here (**8.5.4**). The only Egyptian mummies in Britain are, therefore, those that were brought over by antiquarians and reside in museum collections so care should be taken not to perpetrate misunderstandings and misrepresent Roman mortuary practices in order to gain media attention.

Where eastern influences may be discernible is in the range of resins deemed appropriate for use in the mortuary sphere. Neither Pinaceae nor *Pistacia* spp. resins are mentioned in the primary sources in relation to Roman mortuary practices. They are, however, the most common finds in both the British and continental European burials analysed. This may be due, in part, to their ease of identification and persistence in the archaeological record. Nonetheless, the discrepancy with the literature remains. It is interesting to note, therefore, that *Pistacia* spp. resins were extensively employed in Ancient Egyptian ritual contexts, including as part of embalming mixtures, from the Middle Kingdom to the Roman period (c.f. Buckley & Evershed 2004; Colombini *et al.* 2000; Ménager *et al.* 2014; Serpico & White 2000, 2001; Vieillescazes & Coen 1993). In addition, Pinaceae resins have now been identified in mummy balms as far back as the Prehistoric Period (4500-3350 BC) with a considerable increase in prevalence and the amount applied to the body noted in the Ptolemaic-Roman periods (c.f. Buckley & Evershed 2001; Corcoran & Svoboda 2010; Jones *et al.* 2014; Maurer *et al.* 2002; Koller *et al.* 1998).

With the extension of the Roman Empire to include Egypt, Gaul and finally Britain, this seems to indicate cross-pollination (pun intended) in terms of which resinous exudates were readily available as a result of increased access to resources and knowledge of their efficacy (in both practical and ritual terms) in the mortuary sphere. Thus, Pinaceae resins were making the journey south from Europe to Egypt in far greater abundance with *Pistacia* spp., *Boswellia* spp. and other, as yet to be securely identified, exudates moving in the opposite direction. Thus, Rome sat at the centre of things acting as a melting pot, facilitator and filter for practices deemed appropriate for use in the treatment of the dead.

8.7 Moving towards meaning: reconstructing the fragrant dead

So, can we move beyond their practical aspects and role as markers of status, to consider the ritual significance of these resinous substances? With regards to the plant exudates named or identified, both the literary and archaeological evidence suggests that a limited palette was deemed appropriate for use in the mortuary sphere. A lack of correspondence between the two sources with regards their botanical origins has, however, been noted. This may, in part, be a reflection of losses from the archaeological record due to the chemistry of some of those mentioned in the literature (e.g. gums, phenolic extracts, essential oils; **5.2**). The generic, poetic terminology employed by the Roman authors could also be a factor as *Pistacia* spp. and *L. orientalis*, for example, could be covered by references to balsams and/or the produce of the Levant (**3.2.2**). In addition, changing tastes, eastern influences and access to new markets between the 1st and 4th centuries AD may have come into play (**8.6**) since Pliny refers both to the concept of 'branding' by place of origin in his discussion on perfumes and unguents and to changes in fashionable preferences for certain products (*NH 13.1-2* nd). Only frankincense definitively appears on both lists. This reinforces the well-attested and long-standing ritual importance of *Boswellia* spp. gum-resins in both temple worship and the mortuary sphere which arises from the, seemingly, universal pleasure (human and divine) derived from their aroma (Groom 1981). The recovery of evidence for frankincense (mixed with traces of other exudates) in the Mersea Island cremation urn and, probably, with the cremation (Grave 2) from Purton, confirms the multiplicity of motivations for the inclusion of these scented substances as there could be no practical aspect to their deposition as unburnt offerings.

In the ancient world and throughout human history, different species or genera are known to have been accorded symbolic meanings related to their properties. For example, in the Roman period, bay leaves (*Laurus nobilis*) denoted victory, protection and peace (found with the 'Spitalfields Lady', London, UK), myrtle twigs (*Myrtus communis*), associated with Venus, cleansing and renewal (with two young females, St Maximin's, Trier, Germany) and roses and lilies (*Rosa* spp.; *Lilium* spp.; Grottaferrata, Rome, Italy), love and purity, respectively (e.g. *Pliny 15.36-40* nd; *Virgil 6:708-709, 12:68-69* nd). The properties of significance appear to be a reflection of intrinsic factors such as growth patterns (e.g. evergreen) with colour and fragrance key, especially in terms of plant exudates. Thus, conifer species were associated with immortality and held a special place in Roman funerary symbolism as markers of mourning (Alcock 1980; *Pliny NH 16.18-19* nd; as discussed in **5.3.2**). That Pinaceae resins should not be mentioned in the context of mortuary rites is somewhat surprising but, perhaps, they were too commonplace to warrant comment in poems that sought to emphasise the exotic. Their use in the treatment of the body is, however, clearly supported by the significant quantities employed in embalming during the Roman period in Egypt (c.f. Buckley and Evershed 2001; Colombini *et al.* 2000; Corcoran and Svoboda 2010; Maurer *et al.* 2002). Here, their increased use appears to form part of the changes in mummification techniques which occurred under the influence of first Greek and then Roman overlords with greater emphasis on the outer appearance of the bundle, beautiful wrappings and the provision of portraits, rather than the focus on soft tissue preservation and stylised representations characteristic of earlier Egyptian practices (c.f. Aufderheide 2003: 5-21, 322-394; David 2000).

Much less is known with regards the ritual connotations of *Pistacia* spp. resins in antiquity, particularly in relation to the Roman period. This may be a matter of terminology and taxonomic confusion between the various species with *ela/elah*, Chios turpentine, Cyprus balsam and Balsam of Syria possibly used to denote their exudates (Beckmann 2012; Mills & White 1977; Serpico 1996: 40-54). It was also long thought that the incense, *snṯr*, frequently mentioned in Ancient Egyptian texts, was frankincense although Loret (1949) indicated that mastic/terebinth was more likely. His view was subsequently confirmed by the discovery of an abundance of *Pistacia* spp. resin in Canaanite *amphorae* on the Uluburun shipwreck (Mills & White 1989) and adhering to inscribed sherds from Amarna, Egypt (Serpico & White 2000; Stern *et al.* 2003). The presence of *Pistacia* spp. resins in numerous Egyptian mortuary contexts has now been firmly established and it appears to have been a key ingredient of the balms employed in mummification (e.g. Brettell *et al.* 2015c; Buckley & Evershed 2001, 2004; Clark *et al.* 2013; Colombini *et al.* 2000; Łucejko *et al.* 2012). Likewise, evidence for its exploitation, alongside Pinaceae resins, in Minoan Crete has recently been demonstrated through analysis of residues on ceramics (Beck *et al.* 2008a, 2008b) with its ritual use as part of scented unguents, due to connotations related to overcoming death, proposed on the basis of linguistics and inscriptions on Linear B tablets (Beckmann 2012). In the Old Testament, as the terebinth tree, it again appears in relation to death and burial (e.g. *Bible: SEV* 2009 *Genesis 35:4; I Samuel 17:2; II Samuel 18:9; Chronicles 10:12*).

Thus, although references in classical texts are to its practical and medicinal rather than ritual use (e.g. *Pliny NH 13.2, 16.22* nd; *Theophrastus 9.2:1-2* nd), given the inter-relationships between Rome and these cultural groupings (explored above, **3.2.2; 8.6**), the discovery of *Pistacia* spp. resins in Roman mortuary contexts is not so surprising.

Similar difficulties arise when considering the significance placed on the 'mystery' resin as the term balsam was widely used, including in relation to the treatment of the dead, and almost certainly included a range of botanical sources (*Ausonius Epitaph 6.31* nd; Chioffi 1998: 23; Howes 1949: 153; Langenheim 2003: 356-357; Safrai 1994: 83-87). If the exudate discovered is indeed *Liquidambar orientalis* (storax; **7.5**), classical authors make little reference to its ritual use although it was very highly valued and fragments of its wood, coated with resin, were reportedly burnt as incense (Hafizoğlu 1982; *Pliny NH 12.54* nd; *Theophrastus 9.7:3* nd). That the fragrance produced by cinnamates (present in many balsamic exudates) was strongly associated with the mortuary sphere is, however, clear from the frequent references to cinnamon and cassia (which contain these compounds in abundance) as part of Roman funerary symbolism (**3.2; Table 3.1**).

Whether the different types of cinnamon mentioned (Pharian and Sabaean, from Egypt and Arabia, respectively) derived from 'true' cinnamon (the inner bark of *C. verum*) traded from India via these regions or from a number of otherwise unrelated sources cannot be determined. The latter seems more likely given the varied terminology employed (which implies resinous extracts, woods, leaves and roots were utilised) and the different native flora of these areas with eastern Africa and Syria additionally described as a sources of cinnamon and cassia (*Anon PME 8, 12* nd; *Pliny NH 12.41-43* nd; *Plutarch Lives Sulla* nd). Used with myrrh and nard to build the nest of the Phoenix, substances containing cinnamates seem, therefore, to have held connotations of rebirth with indications for the ritual use of balsamic resins obtained from chemical analysis of Ptolemaic-Roman period mummy balms (Buckley & Evershed 2001; Lucas & Harris 1962: 323; Méjanelle *et al.* 1997) and residues in an incense burner from the Roman/post-Roman necropolis at Antinoe, Egypt (Modugno *et al.* 2006a).

It is also possible that these resinous substances acted not only to demarcate certain individuals as different from the norm in terms of their social status (**8.6**) but were a reflection of other aspects of their identity such as age, gender or affiliation with certain deities. For example *Pistacia* spp. resins are frequently present in the burials of children and young adult females (Arrington infant; child at Anché; 'Spitalfields Lady', London; 'Lady of the Sarcophagus', Milan; Grab 107 and 189B, Trier) although not exclusively (**Table 3.4; Table 8.1**) and a far larger sample would be needed to interrogate such patterns, effectively. Likewise, there may be indications for the differential treatment of areas of the body in terms of the placement of the aromatic exudates selected (**8.5**). At present, this is largely based on the chemical evidence obtained from the multiple contextualised samples of grave deposits from the lead coffin of the 'Spitalfields Lady', London (**7.4**) and so, again, needs further investigation. Nonetheless, there are indications that *Pistacia* spp. resins may have been deposited in greater abundance around the head and upper torso while Pinaceae resins were associated with the pelvic region. That *Pistacia* spp. fragments were found around the cranium of the Arrington infant (**7.2**) while the 'lumps' with 'Lady of the Sarcophagus', Milan were placed on either side of the head and between the legs (Bruni & Guglielmi 2005) may support some form of selective distribution.

Similar patterns have been observed in relation to Egyptian mortuary rites with samples collected from the head of an individual mummified in the 3rd Intermediate Period showing treatment with *Pistacia* spp. resin while the chest cavity had been filled with a Pinaceae pitch (Serpico & White 1998). The need for special treatment of the head, hands, feet and genitals is, in fact, recorded in a number of papyri with associations between the material or mixture employed, its properties (especially colour although texture and smell might also have been significant) and specific deities (Riggs 2010; Serpico & White 2001). It may, therefore, be that in the Roman period, the rich and shining colours associated with death such as red/purple, yellow and white (Bradley 2009: 1-35; Thomas 1979), were similarly materialised through the agency of white garments with gold, purple and/or red decoration alongside floral tributes composed of roses, violets and lilies (c.f. Ghini *et al.* 2005; Reifarth 2013: 47-90; Wild 2013). To this we may now add the selection of rich orange/yellow resins (e.g. mastic/terebinth, storax) and pale cream/white gum- resins (e.g. frankincense).

Thus, it seems, that resinous exudates may have been interred with specific individuals and/or distributed in different areas of the body to provide a ritual text which could be read by those viewing the body, although one which we, at so far a remove, struggle to understand. From the limited data available, the most obvious pattern seems to relate to the importance of the head and its differential treatment in death. This facet of mortuary rites is a well-attested and widespread phenomenon from the plastered skulls of the Neolithic in Anatolia to the beautiful portrait mummies of the Fayum, the creation of death masks and use of *imagines* as part of Roman funerary rites and the cult of the head among the 'Celtic' tribes (e.g. Armit 2006; Bonogofsky 2005; Corcoran & Svoboda 2010; Noy 2011). As demonstrated by theoretical approaches, such concurrences do not arise from a culture-historical style spread from a single source but from common human responses to the natural world and our place within it (Fahlander and Oestigaard 2008; Metcalf and Huntington 1991; Hertz 1960; **2.2**). So, what we may be detecting in these Roman period burials from Britain and Europe is the intermingling of ritual practices among the pre-disposed as indicated by Diodorus Siculus' description of the Celtic practice of preserving the heads of "*distinguished enemies...[for]...exhibit[ion] to strangers*" in cedar oil (*5.29.5* nd). In this context it is interesting to note the void in the plaster body-casings over the faces of some (at least 3) of those interred below St Maximin's, Trier (e.g. Grab 107, **Figure 8.6**). Was this area left open so that the deceased could be viewed? Or was it removed to provide the mould for the creation of a death mask? Either way it adds support to the argument that the head received special treatment in the Roman period.

Likewise, the combination of materials employed in these late Roman ‘resin’ burials may reflect something other than conspicuous consumption. The inclusion of resinous substances, wrapping of the body in textiles and, when present, the addition of plaster (whether gypsum or calcite-based) or wood shavings (a practice continued in the Rhineland until, at least, the 18th century AD; Donoghue *et al.* 2013) would have acted to temporarily slow decay, absorb purgative fluids and/or mask the odour of decomposition. The application of pigments to the skin is also of considerable interest and is mentioned in the literature, with the *pollinctores* so named because they used a fine powder to cover the progressive discolouration of the corpse (c.f. Graham 2011). Thus, all of these components signify attempts to disguise the visual and olfactory impact of corporeal decay, actions which clearly had a practical purpose, but would also have acted to embed a final image of the deceased in the minds of the survivors.

Bedecked in a manner denoting aspects of their identity in this life and/or representing their hopes for the next, this ‘dressing’ of the dead would have enabled an idealised version of the individual (as with stylised depictions on tomb monuments and in epigraphs) to be maintained in memory and enter into eternity. This aim would have been supported by the provision of substantial stone sarcophagi and use of lead-liners or coffins (associated with preservation; *Pliny NH 34.49-54* nd) which afforded additional protection to the body. Thus, all the elements of this ‘package’ appear to be material manifestations of a desire to contain the essence of the individual within an enclosed space. Moreover, it places this rite firmly within the previously identified trajectory towards containment and maintenance of the integrity of the body which began in the late pre-Roman Iron Age and lasted (except for the hiatus created by Anglo-Saxon practices) well into the 20th century AD in Britain and continental Europe (c.f. Esmonde Cleary 1992; Parker Pearson 2003: 41-43; Philpott 1991: 235-240; Whimster 1981: 275; **4.2**).

So, how does this fit with the well-attested tripartite structure of mortuary rites that has been shown to hold true in Roman (and Greek) practice (c.f. Garland 2001: 13; van Gennep 1960; **Chapter 2**)? The evidence compiled during this project indicates that the initial separation of the living and the dead was marked by the application of oils/unguents which were used to cleanse and purify the body. Chemical traces of these complex mixtures of plant oils and extracts containing LMM phenolic and terpenic compounds with, perhaps, a gum or resin as a stabiliser would only be recoverable in exceptional circumstances. Rubbed into the skin, subsequent natural mummification due to specific conditions within the burial environment (e.g. desiccation in an arid, enclosed environment with low microbial activity, formation of adipocere or 'tanning' of the remains as a result of low oxygen, low temperature, moist acidic conditions; Bereuter *et al.* 1996; Chamberlain and Parker Pearson 2001: 83-84) might permit their recovery but, even then, they would probably have had to have been present in considerable abundance.

During the liminal stage, when the deceased was prepared for display, the dressing of the dead appears to have been accompanied by the application of substantial amounts of ritually significant plant products: *"perfumes such as are given to the gods a grain at a time…are piled up in heaps to the honour of dead bodies!"* (*Pliny NH 12.41:83* nd). This array of aromatic exudates, extracts and spices, some of which seem to have been incorporated within the textile wrappings, would have acted to provide an odour of sanctity and retard the activities of microbes and other decomposer organisms. It is these, due to their relative abundance and intimate association with the body, which may be traced in the archaeological record when the individual was inhumed. Finally, during the rites marking the final transformation, offerings were made at the tomb and included scented substances deemed "*an appropriate tribute to the dead"* (*Pliny NH 13.1:3* nd). Clearly demonstrated in the case of the Mersea Island barrow cremation, elsewhere, such evidence may be missed if amorphous masses are no longer visible. When appropriate, the collection of samples not only from the interior (including residues absorbed within the matrix of ceramic cremation vessels) but also the exterior (in case offerings had been scattered within the grave or tomb) of the burial container, regardless of the method of disposal, and the analysis of materials associated with grave goods (especially glass wares and ceramics such as *unguentaria* and *pyxides*) should facilitate the recovery of valuable information (**6.2; 8.5**).

Ultimately, can anything be said about who instigated these actions? Ironically, while undertaking this project, the myriad of practical and symbolic facets entwined in mortuary practices were quite literally brought home as a result of the death of my mother, Gloria June Brettell. As so powerfully demonstrated by Rosaldo (2004), this intermingling of real life and academic research provided a different perspective from which to consider the nature of humanity's responses to corporeal remains. This experience revealed that conflicts may exist between the wishes of the deceased, the needs of the survivors (immediate family and friends) and the demands of society and that these tend to focus around material aspects of the treatment of the body. A similar discrepancy between cultural expectations and individual understandings was encountered by Erasmo during his mother's funeral (2012, xi-xii). In both cases, social norms were victorious resulting in rites that ascribed to dominant regional practices to the emotional discomfort of the living, although of considerable interest as a starting point for investigating the materiality of archaeological mortuary contexts.

What subsequent research has revealed is that, just as with modern mortuary practices and so personally (and uncomfortably) experienced during preparations for the funeral of my mother, the choices made in relation to the treatment of the dead are predominantly driven by social mores. Thus, the evidence compiled here indicates that, over a broad chronological period, members of the Roman elite were provided with a very similar material package with minor modifications to suit the dominant method of disposal. Although acting to disguise the odour of cadaveric decay, the primary role of costly plant products, through their conspicuous consumption on the pyre or deposition in the tomb, was to signify wealth and social status. Heaped around the body on its funeral bed, they were also viewed as offerings made as a mark of the respect and supplication (e.g. *Pliny NH 12.41:83*; *Plutarch Lives Sulla, 38.2* nd; *Statius Silvae 2.1:24-26* nd; **Appendix 1**). Inevitably, it was the survivors who chose to follow this path since, as frequently observed, the dead do not bury themselves, although they may continue to act through the agency of others (c.f. Erasmo 2012: 61-104). Thus, the deceased might have requested certain rites, including specifying which perfumed products were used, as implied in the *Satyricon* where Trimalchio asks to be anointed with nard (*Petronius 77-78* nd). Nevertheless, it was the needs of the survivors to honour (or be seen to honour) their dead and to ensure a successful transition of the spirit of the deceased to the afterlife and their own to the world of the living that were addressed by meeting the expectations of society. They could gain solace in having been seen to have 'done the right thing' even if they had, perhaps, been required to put aside their own conflicting personal preferences.

Finally, as identified in **Chapter 2**, the socially embedded desire for immortality through remembrance appears to have been of considerable importance in the Roman world (c.f. Graham 2011). Part of the powerful, symbol-laden, performance surrounding death, the strongly scented substances used in the treatment of the body would have harnessed the power of smell to trigger recall among the living (and the dead): *"Sprinkle my ashes with pure wine and fragrant oil of spikenard; bring balsam to...Unending spring pervades my tearless urn: I have but changed my state, and have not died. I have not lost a single joy of my old life, whether you think that I remember all or none."* (*Ausonius Epitaph 6.31*). A range of social strategies were, therefore, designed to engage the mourners in individual and collective commemorative acts (e.g. conspicuous funerary processions and public festivals for feasting with the dead). The associated sights, sounds and smells would have had a transformative, cathartic effect, turning death into a celebration, with the greater the sensory impact on the survivors, the more lasting the embedded memory. Thus, through correct action (undertaken by the dying and the survivors) and the agency of materials a 'good death' could be had by all, alleviating the ubiquitous reality of mortality and permitting death to be viewed, not as something to be avoided, but rather as a path to be embraced in the belief that some form of continued existence would follow.

Chapter 9

Conclusion and further work

"We said it was forever but then it slipped away,
Standing at the end of the final masquerade" (Linkin Park 2014)

9.1 Conclusion

The two main hypotheses tested as part of this project were that:

1. resinous substances were transported to Britain during the mid-late Roman period (2nd-early 5th centuries AD) and used in mortuary contexts;
2. these plant exudates, in conjunction with substantial burial containers, plaster body-casings and textiles, held a specific social or ritual meaning.

The bipartite approach employed, which involved the chemical analysis of samples from Roman mortuary contexts and consideration of these findings in relation to theoretical approaches to the materiality of death, enabled both of these hypothesis to be addressed. The analytical data conclusively demonstrated that a limited number of imported plant exudates were used in the mortuary sphere, across the province of *Britannia*, during the Roman period. These comprised European Pinaceae (conifer) resins, *Pistacia* spp. (mastic/terebinth) resins from the Mediterranean or the Levant, eastern African or southern Arabia *Boswellia* spp. (frankincense) gum-resins and, possibly, the balsamic resin derived from *Liquidambar orientalis*. These were present in samples obtained from sixteen of the stone sarcophagus and/or lead-lined inhumation burials assessed and in amorphous residues from two cremation burials. These finds date between the early-mid 2nd and late 4th centuries AD.

One key factor established by this research was the importance, when evaluating mortuary practices, of analysing grave deposits, the mixture of comminuted materials retained in the base of more substantial burial containers. Indeed, it was only by employing a systematic sampling strategy which included such degraded materials that much of the evidence for the presence of resinous substances was obtained. This unprepossessing 'dirt', so often discarded in favour of inorganic artefacts and ecofacts, was found to maintain suites of terpenic compounds long after the breakdown of any visible residues and, where multiple contextualised samples were available (‘Spitalfields Lady’, London; **7.4**), provided indications that deliberate spatial patterning may have been employed in the treatment of the body, with different exudates associated with different regions of the skeletal remains.

Evaluation of the range and relative abundance of the compounds present in these elaborate burials, in conjunction with evidence from an array of primary sources, shows that these aromatic exudates performed multiple roles. Their most mundane function was to disguise the odour of decomposition and aid temporary soft-tissue preservation during the *funus*, when the body was displayed prior to disposal. This aspect was demonstrated, chemically, by the remarkably well-preserved nature of the resinous substances present in many of the inhumation burials (i.e. survival of precursor resin acids, absence/low abundance of markers of extensive degradation or strong heating). This indicated that they had probably been deposited within the grave in their natural state (i.e. unheated) although, in some cases at least, they may have formed part of scented unguents. Found in intimate association with the body and/or incorporated within the textile wrappings, this practice of treating the body with aromatic substances is mentioned by classical authors working in and around Rome at the turn of the first millennium, generally in relation to individuals subsequently cremated. This mortuary practice can, however, now be shown to have extended throughout the Roman period and across the Empire and would have been particularly pressing during the lengthy sequence of funerary rites accorded members of the elite (c.f. Hope 2009, 71-74; **2.3**).

Deposited with both males and females and with individuals of all ages (c. 1 year to >45 years), it also appears that aromatic plant products acted as signifiers of the social standing of the deceased. Their value is reported or implied by many ancient authors who use terms such as ‘costly’ and ‘riches’ to describe these exudates (c.f. *Bible: SEV 2009 Mark 12:3-8*; *Lucan Civil War 8:729-731* nd*; Pliny NH 12.24-59* nd*; Plutarch Sulla 38:2* nd). Indeed, their use in the mortuary sphere seems to be part of a pattern of significant expenditure designed to prevent those of wealth and status suffering the fate of the 'poor man' whose: "*corpse disappears just like his soul*" (*Juvenal Satires 3:260-261* nd). The privileged in society were, therefore, supplied with a burial package which ensured the physical protection of their mortal remains through temporary preservation prior to cremation or inhumation and subsequent indefinite containment, regardless of method of disposal. Alongside the now confirmed use of resinous substances, this additional material investment variously comprised: interment below a barrow or mausoleum, within a glass cremation urn, stone sarcophagus, tile-built structure, lead coffin/liner or ossuary and, on occasion, included being encased in plaster or surrounded by wood shavings.

Moreover, there is now considerable archaeological evidence to support the image, provided by classical authors, of the transportation of the deceased on a funerary bed/bier, wrapped in textiles (shrouds, bandages, face covers) and dressed in costly gender-appropriate garments made from fine linens, wools, silks, with decorative elements picked out in gold thread or using dyes including murex purple (c.f. Wild 2013). Thus, in light of the evidence revealed here, the description of the treatment of Priscilla deserves to be quoted: *"Who could give true account of the rites and gifts of the funeral procession? There, crowded together in a long train, flow the spring produce of Arabia and Cilicia, Sabaean flowers and the flame-feeding Indian harvests, incense carried from Palestinian shrines, and Hebrew essences, Corycian saffron and myrrh; her body lies on a high bier, veiled by silk, and Tyrian purple"* (*Silvae 5.1: 208-215 nd*).

As the terms "*exsequias et dona*" (funerary rites and gifts) implies, the accompanying array of aromatic plant products had yet another role to play in the mortuary sphere. Linked to Greek and, perhaps, Etruscan practices modified by contact with eastern cults, resinous exudates were closely associated with aspects of the divine according them ritual significance across the ancient world. The aroma of sanctity they provided, which was considered so pleasing to the gods that it could carry the essence of offerings to the heavens, would have acted to purify the deceased and facilitate safe passage to the otherworld. Hence, expressions of concern that the dead should not go “*unscented to the urn*” (or grave) since the lack of care and respect implied would have had a detrimental effect on their future existence (*Persius Satire 6:34* nd). Of equal importance, in this regard, may have been the sensory impact of these powerful fragrances on the living. Through the agency of conspicuous consumption these resinous substances, piled around the body to be burnt on the pyre or interred in the tomb, would have contributed to the final exhibition of the munificence and *virtus* of the deceased. This show, enacted by and for the benefit of the survivors, would have served to promote olfactory recall, triggering remembrance of the dead through the private and shared memories of those observing the spectacle, thereby providing a “*semblance of immortality*” (*Cassius Dio 50.24:6* nd).

In relation to Roman Britain, this ritual aspect is confirmed by the recovery of amorphous masses from two cremation urns. Analysis revealed that an abundance of frankincense combined with a lesser amount of Pinaceae resin (and, perhaps, essential oils from other species) had been deposited as an unburnt offering with the ‘ashes’ of a mature adult male at the centre of the Mersea Island barrow. Similarly, traces of *Boswellia* spp. and, possibly, *Pistacia* spp. exudates were incorporated within a mass of fatty matter, probably a saponified plant oil, added to a glass cremation vessel interred in the rural burial ground at Purton, Wiltshire. These finds represent the first chemical confirmation for the inclusion of resinous substances in Roman period cremation burials. What is even more remarkable is that frankincense (possibly from eastern Africa) should have been transported to Britain in such abundance that, as early as the mid 2nd century AD, over 90 g could be included as a mortuary offering on a remote tidal island off the south-east coast. Thus, antiquarian claims for similar practices at Bartlow Hills, Essex-Cambridgeshire borders and Weston Turville, Buckinghamshire (Gage 1834; Waugh 1962) should not be discounted, although which plant exudates were employed can no longer be determined as samples are no longer extant.

The mortuary sphere has always provided an opportunity for socio-cultural display with certain individuals accorded more elaborate rites than the 'norm'. What this research has confirmed, ending centuries of speculation on the subject, is that resinous substances formed such an important element of this Roman elite package that they even reached the remote province of *Britannia*, far from the source of most of the exudates employed. The broader historical significance of this discovery is manifold touching on ritual, socio-cultural and economic aspects of life and death in the Roman world and beyond. The desire for particular aromatic substances which resulted in their transportation from one end of the Empire to the other, particularly at a time when long-distance trade is believed to have been in decline, attests to the importance of these luxury goods. Nonetheless, the precise method of transport, possibly in *amphorae, unguentaria* or *pyxides*, depending on the nature of the exudate (i.e. whether it could be shipped as a viscous liquid, solid fragments or formed part of a scented oil/unguent) and the extent of this trade remains to be ascertained.

Likewise, questions regarding the ethnicity of the individuals interred in this manner and where and when they might have been introduced to this rite have yet to be resolved (isotope analysis of cemetery populations which include individuals interred with and without resinous substances in a variety of containers is required e.g. Alington Avenue and Poundbury, Dorset). Were these scented substances the personal property of migrants, as may have been the case with the 'Spitalfields Lady', London who appears to have been a recent arrival from the vicinity of Rome (c.f. Montgomery *et al.* 2010; **7.4**)? Or were they introduced by members of the native elite who had travelled abroad (as merchants, administrators, soldiers) and were keen to display their acquired *romanitas*, as may have been the case with the mature male from Mersea Island due to the composite nature of his barrow burial (**7.11**)? Or, by the late Roman period (to which most of the British finds date; **Table 8.1**), had knowledge of this practice become so embedded in the consciousness of the inhabitants of Britain that the exudate or unguent of choice could simply be selected, alongside other material manifestations of wealth and status, from a range of options supplied by the local *libitinarii*?

Our last performance in this world, the period between death and burial affords the final sensory impact of the deceased upon the living. Mortuary rituals performed during this liminal phase must deal with the biological reality of the decomposing body but are also crucial in renegotiating the structure of society. Through enactment of the correct, culturally-situated, processes transference of the spirit of the deceased to the otherworld is navigated so that the survivors may return their transformed lives within the re-balanced social order (c.f. Hertz 1907; van Gennep 1909). Thus, adorned to enter the next world in a manner emblematic of their idealised status in this life (as inversions must not be forgotten), the materiality of resinous substances would have served to imprint a final image of these members of the Roman period elite in the minds of the living and help play out the final masquerade.

9.2 Further work

As this work progressed, questions raised led to ancillary research projects (**Appendix 7.6**) and revealed a number of other avenues that remain to be explored. Hopefully, future projects based on strategic sample collection from newly excavated sites will enable our knowledge of this Roman mortuary practice to be expanded, with the analysis of fully contextualised grave deposits (x10-20 spatially distributed <5 g samples plus x2-3 controls) from sarcophagus and/or lead-lined coffin burials recommended (8**.2; Appendix 2.2**). This approach would not only permit resinous substances present to be characterised but would also to enable more intricate questions to be addressed. For example, the possibility of spatial patterning in relation to different areas of the body as highlighted by variations in the relative abundance of the two resins applied to 'Spitalfields Lady'. Extending this research to systematically evaluate suites of burials, from mausolea, stone sarcophagi and/or lead-lined coffins alongside more normative inhumations in other provinces of the Roman Empire, such as the various regions of Gaul and Hispania, even Italy itself, would be beneficial in developing our understanding of this rite. In addition, analysis of residues adsorbed within the fabric of ceramic cremation vessels might be worth investigation.

Consideration of the method by which these plant exudates were transported and exploration of their wider use in the ritual sphere during the Roman period would then help situate these mortuary practices within their broader socio-economic context. Ideally this would involve the analysis of residues from glass and ceramic vessels (e.g. *unguentaria, pixies, amphorae*) as well as house shrines, temples and storage areas. This would probably prove a considerable challenge in terms of Roman Britain due to the near absence of securely identified temple sites and the detrimental impact of environmental factors on the survival of chemical evidence for plant exudates in unprotected contexts (due to comminution, bioturbation, water movement and intermingling with soil organic matter). Nonetheless, as resinous substances have been recovered from warehouses, settlement sites and shrines in more arid regions (c.f. Evershed *et al.* 1997; Mathe *et al.* 2007; Regert *et al.* 2008) it might, again, be advisable to extend this search to finds and sites from continental Europe.

Depending on the research agenda, a tighter analytical focus on recovering evidence of terpenic compounds rather than or, preferably, in addition to the generic approach taken here might prove valuable. This could be achieved through the use of single ion monitoring (SIM) and the targeting of key fragment ions (e.g. *m/z* 121, 241, 256, 359 for diterpenoids; *m/z* 189, 203, 218, 292 for triterpenoids) which would assist in elucidating the nature of any resin acids present and some of their derivatives. It could also bring to light components that would have fallen below the limits of detection in full-scan mode due to the increased dwell time permitted by limiting the number of ions detected. Nonetheless, evidence regarding other interesting substances might be lost (e.g. phenolic compounds, markers of fats/oils or bitumen) if this approach was exclusively employed.

The suggestion that the balsamic resin indicated by the combination of compounds in the focal burial from the Eagle Hotel site, Winchester, a number of sarcophagus burials from Trier and, perhaps, the ‘headband’ from Poundbury comprised the exudate of *Liquidambar orientalis* also requires testing. A series of laboratory-based degradation experiments has been offered as a 4th year Chemistry and Forensic Sciences project, University of Bradford, Bradford, UK. This research will, it is hoped, ascertain if the terpenoids present in fresh extracts (i.e. the 3α-epimers) can be epimerised (to the 3β-epimers) by the combination of an Oppenauer oxidation and a Meerwein-Ponndorf-Verley reduction prompted, perhaps by the presence of yeast or bacteria and/or catalysed by various metal ions that may have been present in the burial environment (e.g. lead, calcium, sodium). Likewise, as little is known with regards the natural degradation pathways of the compounds present in frankincense slow chemistry experiments would be invaluable in assessing the impact, over time, of different factors on this gum-resin.

Finally, the methodology developed here could clearly reap rewards if targeted at the identification of substances used in the treatment of the body as part of mortuary practices from other archaeological/historical periods and regions of the globe. In relation to this research agenda, it would be particularly interesting to consider evidence from Greek, Etruscan and Jewish mortuary contexts in order to establish precursors for this Roman rite. It would also be fascinating to investigate its medieval successors, for example, the possibility that the ‘uncorrupted’ bodies of saints and martyrs resulted from the continued application of this knowledge and that resinous substances contributed to the production of relics.

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THE FINAL MASQUERADE: A MOLECULAR-BASED APPROACH TO THE IDENTIFICATION OF RESINOUS PLANT EXUDATES IN ROMAN MORTUARY CONTEXTS IN BRITAIN AND EVALUATION OF THEIR SIGNIFICANCE

Volume II of II

Rhea Catharine BRETTELL

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Appendix 1. Primary sources and plant exudates

Texts from the 2nd century BC to the 6th century AD containing references to anointing and use of aromatic substances in the treatment of the dead

1.1 Anointing

Source: *Ennius Annales 3:157*
Date: 2nd century BC
Genre: Epic poetry – the death of Tarquin
Translation: Warmington 1935 http://www.attalus.org/poetry/ennius1.html

"the good woman washed and anointed the body of Tarquin"

Source: *Virgil Aeneid 6:218-220*
Date: 1st century BC
Genre: Epic poetry – preparations for the cremation of Misenus, a Trojan
Translation: Hope 2007: 97

"they washed and anointed the cold body of their friend and lamented. When they had wept enough, they placed him on the bier and covered him with his own purple robes".

Source: *Ovid Fasti 4:851-853*
Date: 1st century BC
Genre: Calendar – religious festivals of Rome - Romulus with Remus's body
Translation: Kline 2004 http://www.poetryintranslation.com

"When they set down the bier, he gave it [the body] a last kiss,
And said: 'Farewell, my brother, taken against my will!'
And he anointed the body for burning".

Source: *Propertius Elegies 2.13b:13-16*
Date: 1st century BC
Genre: Elegiac poetry
Translation: Katz 2004: 127

"You will place your final kisses on my icy lips,
"When the alabaster jar full of Syrian oils will be offered.
Then, when the flame has been applied and turned me into ash,
Let a small jug receive my remains".

Source: *Petronius Satyricon 77-78*
Date: 1st century BC
Genre: Satirical novel – Trimalchio makes his dinner guests enact his funeral
Translation: Hope 2007: 119

"bring me the clothes in which I mean to be carried out. And some ointment, and a sample from that jar which is to be poured over my bones...At once he opened a jar of ointment and anointed us all and said, 'I hope that I shall like this as much when I'm dead as I do when living'. Besides this he ordered the wine to be poured into a bowl, and said, I want you to think that you've been invited to the Parentalia".

Alternative translation: Sullivan 1977: 88

"'bring out the shroud and the things I want to be buried in. Bring some cosmetic cream too, and a sample from that for of wine I want my bones washed in'...He opened a bottle of nard on the spot, rubbed some on all of us and said: 'I hope this'll be as nice when I'm dead as when I'm alive'. The wine he had poured into a big decanter and he said: 'I want you to think you've been invited to my wake".

Source: *Persius Satire 3:104-105*
Date: Mid 1st century AD
Genre: Satirical poetry
Translation: Ramsey 1918: 353-354

"the...departed, laid out on a high bed and smeared with greasy unguents [amomis], stretches out his heels cold and stark towards the door"

Alternative translation: Carroll 2011: 130

"poor fellow....all made up with powder and some sweet smells high on a bier with feet turned towards the door"

Source: *Martial Epigrams 5:64*
Date: Late 1st century AD
Genre: Poetry
Translation: Hope 2007: 52

"Pour in, Callistus, two double measures of Falerian [wine];
And Alcimus dissolve the summer's snow;
Let my wet hair be rich with excessive amomum,
And my temples weary beneath the woven roses.
For the mausolea close by command us to live,
For they teach us that the very gods can die."

Source: *Juvenal Satire 4:108*
Date: Late 1st-early 2nd century AD
Genre: Satirical poetry
Translation: Ramsey 1918: 65

"reeking at early dawn with odours [amomo] enough to out-scent two funerals"

Source: *Lucian On Funerals (Of Mourning):11-15*
Date: 2nd century AD
Genre: Satirical essay
Translation: Hope 2007: 99

"Then they wash them...Having anointed the body, which is already speeding to decay, with fine perfumes, and crowning it with beautiful flowers, they lay the dead in state, dressed in splendid clothes...the dead man, calm and handsome, elaborately garlanded with wreaths, lies in a lofty and exalted state, decorated as if for a pageant"

Alternative translation: Fowler & Fowler 2007: 214-215

"the corpse is next washed, anointed with the choicest unguents to arrest the progress of decay, crowned with fresh flowers and laid out in sumptuous raiment...he, bravely attired and gloriously garlanded, reposes gracefully upon his lofty bier, adorned as it were for some pageant"

1.2 Part of the funeral procession

Source: *Propertius Elegies 2.13b:3-8*
Date: 1st century BC
Genre: Elegiac poetry
Translation: Katz 2004: 127

"Let there be no long procession of my family portraits, no trumpet bewailing my fate in vain. Let there be no bier spread with ivory headrest, nor let my corpse lie on an Attalic couch. Let there be no array of delicious platters; but rather the modest rites of a common funeral."

Alternative translation: Hope 2007: 90

"Let there be no procession of incense bearers, but only the humble rites that mark a poor man's funeral".

Source: *Plutarch Sulla 38:2*
Date: 1st century AD
Genre: Biography
Translation: Perrin 1916: 444

"And it is said that the women contributed such a vast quantity of spices for it, that, apart from what was carried on two hundred and ten litters, a large image of Sulla himself, and another image of a victor, was moulded out of costly frankincense and cinnamon".

Source: *Statius Silvae 2.1:24-26*
Date: Late 1st century AD
Genre: Poetry – lament for Glaucias, the favourite foster son of Melior
Translation: Nagle 2004: 66-67

"the grim procession's solemn rites, the boy's atrocious bier, watched by the City; I saw the cruel accursed incense heaped, the spirit wailing over its own corpse".

Source: *Statius Silvae 5.1:208-215*
Date: Late 1st century AD
Genre: Poetry - in memory of Priscilla, wife of Abascantus
Translation: Shackleton Bailey 2003: 327-330

Who could give true account
Of the rites and gifts of the funeral procession? There, crowded
Together in a long train, flow the spring produce of Arabia and
Cilicia, Sabaean flowers and the flame-feeding Indian harvests,
Incense carried from Palestinian shrines, and Hebrew essences,
Corycian saffron and myrrh; her body lies on a high bier, veiled
By silk, and Tyrian purple".

Alternative translation: Nagle 2004: 156-157

"Who could compose a song worthy of her funeral procession, the gifts which that unfortunate cortege offered her corpse? Crammed in that swarming length what one whole spring gave Arabs and Cilicians flowed by, flowering Sabaean frankincense, the Indian crop for burning, incense robbed from Palestine's temples, also the balm of Gilead, threads of Corycian saffron, and beads of myrrh, while she herself reclines on high bolsters covered in Chinese silk beneath the shade a purple canopy provides."

1.3 Placed on the pyre or, perhaps, in the tomb

Source: *Propertius Elegies 4.7:32-34*
Date: 1st century BC
Genre: Elegiac poetry
Translation: Katz 2004: 387

"Why didn't my flames smell of spikenard? Was that so hard, to toss hyacinths, which cost nothing, to purify my ashes with the broken jug?"

Source: *Pliny Natural History 12.41:83*
Date: 1st century AD
Genre: Natural History
Translation: Rackham 1968: 61

"[Arabia's] good fortune has been caused by the luxury of mankind even in the hour of death, when they burn over the departed the products which they had originally understood to have been created for the gods. Good authorities declare that Arabia does not produce so large a quantity of perfume in a year's output as was burned by the Emperor Nero in a day at the obsequies of his consort Poppaea. Then reckon up the vast number of funerals celebrated yearly throughout the entire world, and the perfumes such as are given to the gods a grain at a time, that are piled up in heaps to the honour of dead bodies!"

Alternative translation: Hope 2007: 112

"Luxury has made them [the products of Arabia] sacred even at people's deaths. They are thought to have been made by the gods for burning with the dead. Those knowledgeable about the matter have said that more than a year's supply was burned by the Emperor Nero on the last day of his wife Poppaea. It is estimated that throughout the entire world every year as much is given at funerals, heaped, and piled in honour or the corpse, as is given little by little to the gods".

Source: *Lucan Civil War 8:729-731*
Date: Mid 1st century AD
Genre: Epic poem – funeral of Pompey in 48 BC who was murdered in Egypt
Translation: Duff 1928: 491

"No costly
pyre with heaped-up incense does your favourite,
Pompey, ask of you. Fortune; he does not ask that
the rich smoke should carry to the stars Eastern
perfumes from his limbs"

Alternative translation: Hope 2007: 111

“He does not request a costly pyre heaped with incense to give off a rich smoke of eastern perfumes to waft up to the stars”

Source: *Martial Epigrams 10:97*
Date: Late 1st century AD
Genre: Poetry
Translation: Hope 2007: 113

“When the insubstantial pyre was being built with papyrus that was soon to be burned and his tearful wife was buying myrrh and cassia.”

Source: *Martial Epigrams 11:54*
Date: Late 1st century AD
Genre: Poetry
Translation: Hope 2007: 113

“Shameless Zoilus, empty your dirty pockets of the unguents and the cassia and the myrrh smelling of funerals and the half-cremated frankincense you took from the pyre and the cinnamon you snatched from the Stygian couch”

Source: *Statius Silvae 2:4.43-46*
Date: Late 1st century AD
Genre: Poetry – on the death of Melior’s parrot
Translation: Nagle 2004: 83

“With spicy Eastern oils the corpse is burned, while fine and dainty feathers exhale the scent of myrrh from Arab lands and Sicily’s saffron”.

Alternative translation: Mozley 1928: 115

“his ashes are rich with Assyrian balm [amomo], and the frail feathers breathe incense of Arabia and Sicanian saffron”

Source: *Statius Silvae 2.6:85-88*
Date: Late 1st century AD
Genre: Poetry – lament for a favourite slave of Flavius Ursus
Translation: Hope 2007: 112

“The fire consumed fragrant forests of incense and saffron, and cinnamon stolen from the Phoenix, and the juices that drip from Assyrian herbs, as well as your master’s tears”

Alternative translation: Mozley 1928: 127

“fragrant harvests of Saba and Cilicia did the fire consume, and cinnamon stolen from the Pharian bird, and the juices that drip from Assyrian herbs – and thy masters’ tears”

Source: *Statius Silvae 3.3:42-48*
Date: Late 1st century AD
Genre: Poetry – consolation to Claudius Etruscus on the death of his father
Translation: Nagle 2004: 106-107

“You, flood the pyre, Etruscus, with lavish Eastern balm, you, flood it now with grand harvests – Cilician ones of saffron, and Arabs’ incense. Let the fire take off your whole inheritance; mound up your gifts on ashes that will send devoted clouds of smoke to shining heaven”.

Alternative translation: Mozley 1928: 171

“Do thou with lavish hand plunge Eastern incense in the flames, and the proud harvests of Cilicia and Araby; let the fire consume thy heritage of wealth”

Source: *Statius Silvae 2.1:214-227*
Date: Late 1st century AD
Genre: Poetry – lament for Glaucias, the favourite slave child of Melior
Translation: Nagle 2004: 71

“the funeral liturgy, the lavish gifts the flames receive, the body set ablaze with mournful opulence, the fact a mound of purple rugs enlarge your gloomy pyre, the fact that choice Cilician saffron bathed the hair which soon would blaze, and Indian spice, and perfumed oils that came from everywhere, Arabian myrrh, Egyptian cinnamon, and balm of Gilead?”

Alternative translation: Mozley 1928: 89

“heap the purples high on the sad pile, how Cilician blooms and gifts of Indian herbs, and juices of Arabia and Palestine and Egypt steeped the hair that was to burn?”

1.4 Added to the remains after cremation

Source: *Tibullus Elegies 1.3:5-8*
Date: 1st century BC
Genre: Elegiac poetry – illness in Phaecia
Translation: Hope 2007: 115

"Black death keep away, I pray. I have no mother here to gather up the burned bones to her grieving breast; no sister to put Assyrian perfumes on my ashes and weep beside the grave with dishevelled hair."

Source: *Pseudo-Tibullus (Lygdamus) Elegies 3.2:15-26*
Date: 1st century BC
Genre: Elegiac poetry
Translation: Williams 1908 http://www.gutenberg.org

"Then call upon my ghost with holy prayer, and pour ablution o'er their fingers pale....sprinkle with rare, old wine, and gently cast iIn bath of snowy milk, with pious care. These will they swathe with linen mantles o'er, and lay unmouldering in their marble bed; then gift of Arab or Panchaian shore, Assyrian balm and Orient incense shed."

Source: *Persius Satire 6:34-36*
Date: Mid 1st century AD
Genre: Satirical poetry
Translation: Ramsey 1918: 395-397

"he will stint the funeral feast, and will commit your bones unscented to the urn, not caring to enquire whether the cinnamon has lost its fragrance or the cassia has been adulterated with cherry."

Source: *Ausonius Epitaphs 6.31 "On the tomb of a happy man"*
Date: 4th century AD
Genre: Poetry – epitaphs on the heroes who took part in the Trojan War
Translation: Evelyn White 1919: 159

"*Sprinkle my ashes with pure wine and fragrant spikenard; bring balsam too, O stranger, with crimson roses. Unending spring pervades my tearless urn: I have but changed my state, and have not died. I have not lost a single joy of my old life, whether you think that I remember all or none."*

1.5 Methods of disposal

Source: *Lucretius On the Nature of Things 3:886-893*
Date: Mid 1st century BC
Genre: Philosophical (Epicurean) poetry
Translation: Hope 2007: 107

"For if it is really an evil after death to be mauled by the jaws of animals, I cannot see why it is not painful to roast in the hot flames of a funeral pyre, or to lie embalmed in honey, stifled and stiff with cold, on top of an icy rock, or to be crushed under a heavy weight of earth"

Source: *Cicero Tusculan Disputations 1.45:108*
Date: Mid 1st century BC
Genre: Philosophical dialogue
Translation: Hope 2007: 107

"The Egyptians embalm their dead and keep them in the house; the Persians even cover them with wax before burial, so that the bodies may last for as long as possible; it is the custom of the Magi not the bury the bodies of their dead unless they have first been torn to pieces by wild animals"

Source: *Lucian On Funerals (Of Mourning):21*
Date: 2nd century AD
Genre: Satirical essay
Translation: Fowler & Fowler 2007: 217

"The Greeks burn their dead, the Persians bury them; the Indian glazes the body, the Scythian eats it, the Egyptian embalms it"

1.6 Embalming

Source: *Tacitus Annales 16.6*
Date: Late 1st century AD – describing embalming of Poppaea in AD 66
Genre: History
Translation: Grant 1971: 384

"Soon after the Games Poppaea died....She was buried in the Mausoleum of Augustus. Her body was not cremated in the Roman fashion, but was stuffed with spices and embalmed in the manner of foreign potentates".

Alternative translation: Hope 2007: 110

"The body was not cremated in the Roman fashion, but in the tradition of foreign courts was embalmed by stuffing with spices and then was interred in the Julian mausoleum"

Source: *Statius Silvae 5.1:225-231*
Date: Late 1st century AD
Genre: Poetry - in memory of Priscilla, wife of Abascantus, freedman of Domitian
Translation: Shackleton Bailey 2003: 327-330

....Here your peerless consort laid
You to rest, Priscilla, beneath a dome, gently covering you with
Rich Sidonian purple (since he could not tolerate the sound and
Smoke of a pyre). Age will no longer wither you, nor will the
Effects of the years do you harm: such care is taken of your body,
Such are the riches that the venerable marble breathes.

Source: *Cassius Dio Roman History 51.11*
Date: Late 2nd century AD
Genre: History – suicide of Mark Antony in Alexandria, Egypt in 30 BC
Translation: Cary 1917: 31-33

"So Antony died there in Cleopatra's bosom...After this they put out of her way everything by means of which she could cause her own death and allowed her to spend some days where she was, occupied in embalming Antony's body"

Source: *Corippus In laudem Justini Augusti minoris 3.22:27*
Date: 6th century AD
Genre: Eulogy – the death and funerary rites of the Byzantine Emperor, Justinian I
Cited by: Chioffi 1998: 23

Justinian who died in Constantinople was embalmed with honey, balsams, unguents and incense.

1.7 Miscellaneous

Source: *Pliny the Younger Letters 54 (To Marcellinus):16*
Date: 1st century AD
Genre: Letters – writing about the death of a friend's young daughter
Translation: Radice 1969: 152

"No words can express my grief...for the money he had intended for clothing pearls and jewels [for her wedding was now] to be spent on incense, ointment and spices"

Source: *Plutarch Moralia 5.4*
Date: 1st century AD
Genre: Essays – consolatory letter to his wife on the death of their daughter
Translation: Shilleto 1898: 87 http://www.gutenberg.org

"And how unreasonable is it, as some husbands do, to quarrel with their wives about perfume and purple robes, while they allow them to shear their heads in mourning, and to dress in black, and to sit in idle grief, and to lie down in weariness!"

Source: *Josephus Antiquities of the Jews 14.7:4*
Date: 1st century AD
Genre: History – the arrival of Crassus in Judea and death of Aristobulus II
Translation: Whiston 1828 http://www.archive.org

"Pompey's party...destroyed him [Aristobulus] by poison; and those of Caesar's party buried him. His dead body also lay, for a good while, embalmed in honey, till Antony afterward sent it to Judea, and caused him to be buried in the royal sepulcher."

Source: *Josephus The Wars of the Jews, 1.9:1*
Date: 1st century AD
Genre: History – the death of Aristobulus II at the hands of Pompey's followers
Translation: Whiston 1828 http://www.archive.org

"*he was taken off by poison given him by those of Pompey's party; and, for a long while, he had not so much as a burial vouchsafed him in his own country; but his dead body lay, preserved in honey, until it was sent to the Jews by Antony, in order to be buried in the royal sepulchers."*

Source: *Statius Silvae 5.3:53-59*
Date: Late 1st century AD
Genre: Poetry – lament for his father
Translation: Nagle 2004: 166

“Here I lament for you with funeral gifts and eulogy...(no sweeter scent comes from Sicilian saffron, nor rich Sabaeans’ special cinnamon picked just [for you], nor from the Arabs’ crop, their fragrant frankincense)”.

1.8 Jewish sacred texts (Hachlili 2005: 383-385)

There is limited evidence in the Rabbinic literature for Jewish burial rites. The only details come from the minor tract, the Semahot, in the Babylonian Talmud (8th-9th century AD) which is based on two earlier sources the Mishnah (c. 200 AD) and Tosefta (c. 300 AD).

Perfumes should be sprinkled on the body of the deceased (*Babylon Talmud, Sanhedrin 48b*)

The body should be cleaned, purified and anointed with water and oil in preparation for burial before being wrapped in shrouds (*Mishnah Shabat 23:5*)

The bones may be sprinkled with wine and oil...dried herbs may be put on them (*Semahot Tractate 12:9*).

The lamp was not to be lit but to honour the dead and the perfumes were not to be smelled but to prevent the odour of death (*Babylonian Talmud, Brachot 53A*): “*the work of the spices is to remove the odour*”.

1.9 Biblical texts

Source: *Old Testament Exodus 30:22-38*
Date: 6th-4th century BC
Genre: Sacred text – instructions from God on making the holy anointing oil
Translation: SEV 2009

"The LORD spoke to Moses, ' Take the finest spices: of liquid myrrh 500 shekels, and of sweet-smelling cinnamon half as much...and 250 of aromatic cane [calamus], and 500 of cassia...and a hin of olive oil. And you shall make of these a sacred anointing oil blended as by the perfumer; it shall be a holy anointing oil...' And you shall say to the people of Israel, 'This shall be my holy anointing oil throughout your generations...' And the LORD said to Moses, 'Take sweet spices, stacte [myrrh] and onycha and galbanum, sweet spices with and pure frankincense (of each shall there be an equal part), and make an incense blended as by the perfumer, seasoned with salt, pure and holy...you shall not make any for yourselves...It shall be for you holy for the LORD. Whoever makes any like it, to use as a perfume shall be cut off from his people.'

Source: *New Testament Mark 14:3-8*; similarly *Matthew 26:6-12*
Date: 1st century AD
Genre: Sacred texts – events prior the crucifixion of Jesus
Translation: SEV 2009

"as he was reclining at table, a woman came with an alabaster flask of ointment of pure nard, very costly, and she broke the flask and poured it over his head...But Jesus said,...she has done a beautiful thing to me...she has anointed my body beforehand for burial".

Source: *New Testament Mark 16:1*; *Luke 23:56-24:1*; *John 19:39-40*
Date: 1st century AD
Genre: Sacred texts – events after the crucifixion of Jesus
Translation: SEV 2009

"When the Sabbath was past, Mary Magdalene, Mary the mother of James, and Salome bought spices, that they might come and anoint Him." Mark 16:1

"Then they returned and prepared spices and ointments. On the Sabbath they rested according to the commandment. But on the first day of the week, at early dawn, they went to the tomb taking the spices they had prepared." Luke 23-24

"Nicodemus also, who earlier had come to Jesus by night, came bringing a mixture of myrrh and aloes, about seventy-five pounds in weight. So they took the body of Jesus and bound it in linen cloths with the spices, as is the burial custom of the Jews" John 19:39-40

1.10 Early Christian texts

Source: *St Augustine Sermon 177: 7*
Date: 4th century AD
Genre: early Christian theology
Translation: author

"If a rich man is buried with perfumes he may delay bodily corruption but will he not still decay?"

Source: *John of Chrysostom Homilia 85*
Date: 4th century AD
Genre: Homily – on chapters from the gospel of John about the death of Christ
Translation: Marriott 1841 http://www.archive.org

"And they brought those spices whose special nature is to preserve the body for a long time and not to allow it quickly to yield to corruption...[they] buried him, not as a criminal, but magnificently, after the Jewish fashion, as some great and admirable one".

"John tells us...that [the body]...was buried with much myrrh, which glues linen to the body".

"for what is the meaning of that superfluous and unprofitable expense, which brings much loss to the mourners, and no gain to the departed...for the costliness of burial has often caused the breaking open of tombs...[although to prevent this the bereaved often]...cut in pieces those fine clothes, and filled them with many spices...[but even if]...the body itself remains untouched until the Resurrection...what advantage is there from this...when the body is raised naked"

"this corruptible and earthy body shall put on a garment more glorious than silk or cloth of gold, the garment of immortality"

Source: *Prudentius Cathemerinon 10:169-172*
Date: 4th century AD
Genre: Christian hymn – for the burial of the dead
Translation: Pope 1905 http://www.gutenberg.org

"But we will honour our dear dead with violets and garlands strown, and o'er the cold and graven stone shall fragrant odours still be shed."

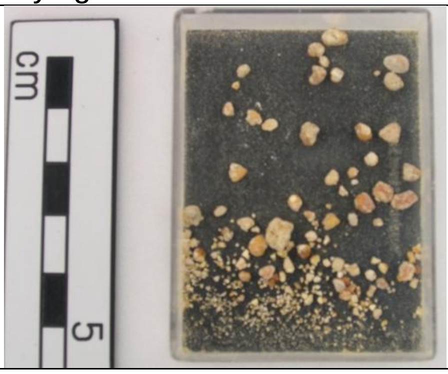
Appendix 2. Methodological protocols

Sample and analytical protocols employed as part of this project

2.1 Sample recording form

Date collected		**Sample no.**	
Site		**Grave**	
Date		**Context**	
Acc. No.		**Reference**	
Context details			
Outer		**Inner**	
Packing materials		**Grave goods**	
Textiles		**Other**	
Body condition		**Body position**	
Sex		**Age**	
Pathologies			
Sample taken			
Images			
Description			
Storage history	In plastic bag & cardboard box	**Sampling procedure**	
Comments			

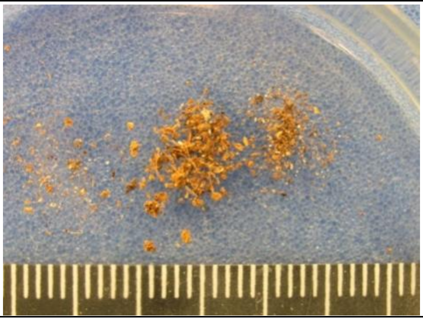
Example: visible fragments with infant burial, Arrington, Cambridgeshire

Date collected	5th December 2011	**Sample no.**	ARR
Site	Arrington, Cambridgeshire	**Grave**	Isolated inhumation
Date	2nd-4th century	**Context**	-----------
Acc. No.	MAA 1994.19	**Reference**	Taylor 1993
Context details	Discovered by workmen during laying of a sewage pipe by East Anglian Water		
Outer	Oak	**Inner**	Lead
Packing materials	Lime or lead ? carbonate	**Grave goods**	Wooden box on lid at foot with pipeclay figurines
Textiles	Fragments of dyed wool	**Other**	Small dense mass of matter hair
Body condition	Cranium – good Post-cranial – poor	**Body position**	Orientated west-east
Sex	-----	**Age**	c. 12 months
Pathologies	Bilateral *cribra orbitalia* Not hydrocephalic as reported		
Sample taken	4 fragments of varying size and colour		
Images			
Description	Numerous opaque yellow-orange fragments Found around the cranium of the infant Brittle with an aromatic fragrance when crushed		
Storage history	Plastic container	**Sampling procedure**	Collected using tweezers Placed in glass vial
Comments	Powdered easily when handled		

Example: control, outer surface of plaster, B8, Poundbury, Dorchester

Date collected	29th May 2012		**Sample no.**	PD8 G1o
Site	Poundbury, Dorchester, Dorset	**Grave**	PD8	
Date	?	**Context**	Main cemetery	
Acc. No.		**Reference**	Farwell & Molleson 1993	
Context details	Main late Roman cemetery R2 mausoleum			
Outer	Ham Hill	**Inner**	None	
Packing materials	Gypsum	**Grave goods**	Fragments of bone comb near cranium	
Textiles	Impressions	**Other**		
Body condition	?	**Body position**	Standard	
Sex	---	**Age**	---	
Pathologies	?			
Sample taken	Layer removed from outer surface of gypsum			
Images				
Description	Layer taken from outer surface of gypsum to provide background soil contamination control sample			
Storage history	In plastic bag & cardboard box	**Sampling procedure**	Scrapped with spatula	
Comments	Excavated 1967 and reburied in situ			

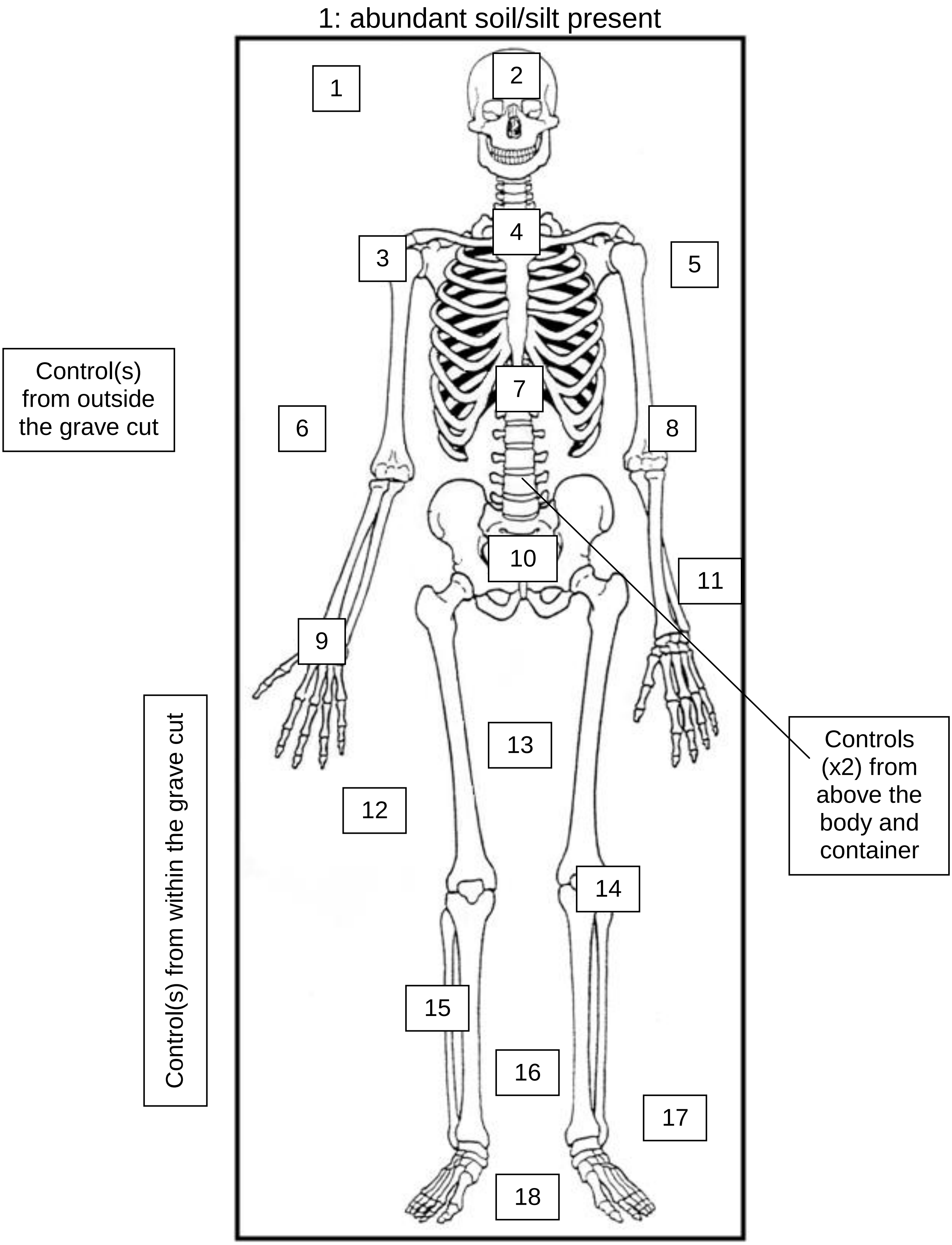
Example: residue, mandible, child, G4378, Alington Avenue, Dorchester

Date collected	30th May 2012	**Sample no.**	AAm
Site	Alington Avenue, Dorchester	**Grave**	4378 (SK1169) context 764
Date	3rd century	**Context**	Small rural burial ground
Acc. No.	1991.89.34	**Reference**	Davies *et al.* 2002
Context details	Rural, boundary edge cemetery, near small farmstead, by road to SE of Dorchester, 1st-4th century AD burials		
Outer	Wood	**Inner**	Lead
Packing materials	None	**Grave goods**	Black Burnished Ware jar; coin of Marcus Aurelius, iron rod
Textiles	Tunic with Tyrian purple wool border	**Other**	
Body condition	Good	**Body position**	Extended; head to the SE
Sex	?male	**Age**	4-6 years
Pathologies	None		
Sample taken	Residue from mandible		
Images	 		
Description	Dark residues adhering to and staining the mandible		
Storage history	In plastic bag & cardboard box	**Sampling procedure**	Removed with blunt end of small spatula
Comments	Very good organic preservation		

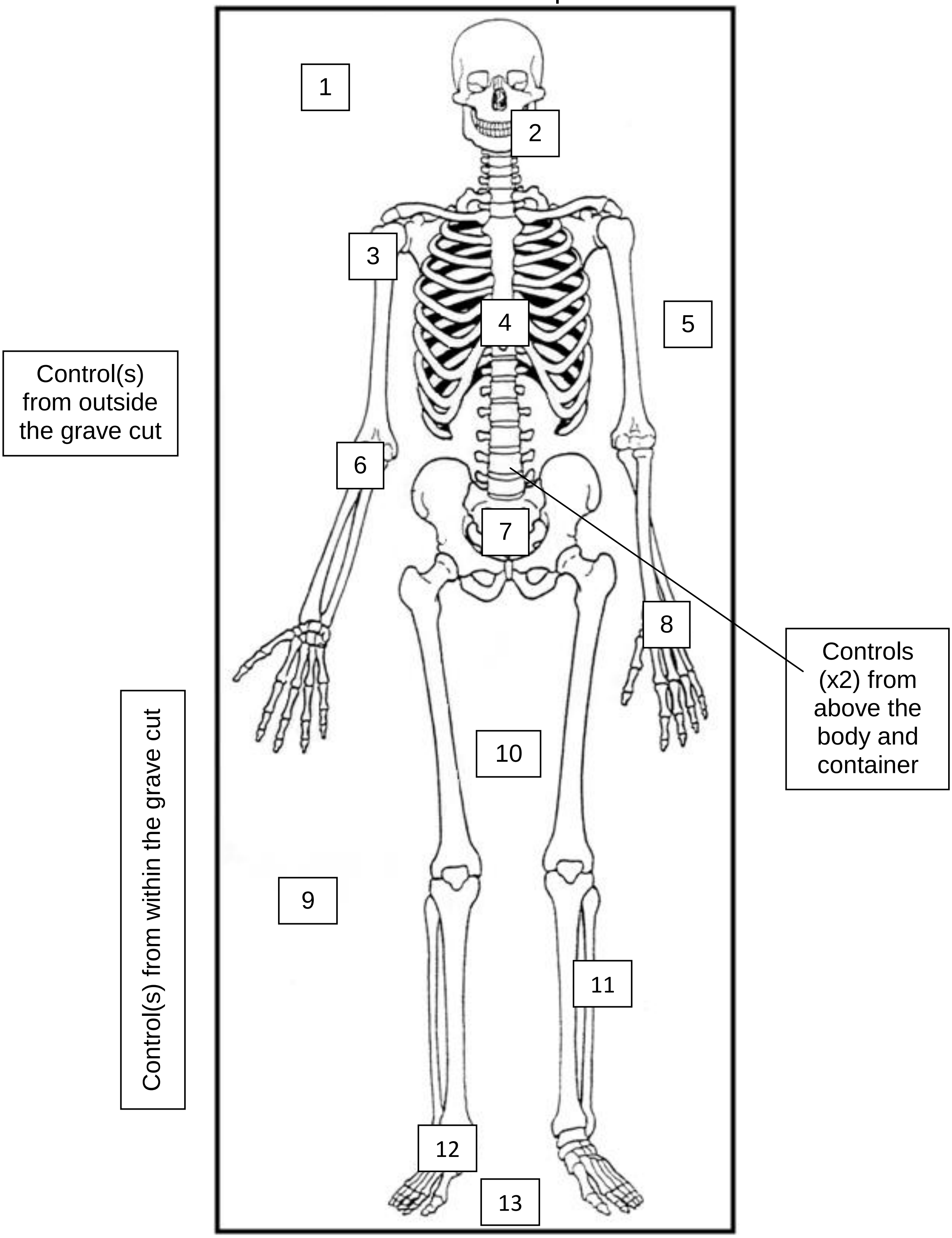
Example: grave deposits, base of lead coffin, Spitalfields Lady, London

Date collected	22nd Jan 2013	**Sample no.**	SPF P12
Site	280 Bishopsgate, London, E1	**Grave**	'Spitalfields Lady'
Date	Mid-4th century	**Context**	North cemetery, London
Acc. No.	SRP 98; SK15903	**Reference**	Thomas 1999; *et al.* 2003
Context details	Part of row of 5 high status burials placed on slight rise at right angles to Ermine Street		
Outer	Barnack limestone	**Inner**	Lead - decorated
Packing materials	None	**Grave goods**	x2 glass phials, jet rod, jet artefacts, box
Textiles	'Pillow' + silk damask & wool	**Other**	'Pillow' of bay leaves; gold thread
Body condition	Well preserved	**Body position**	Supine; head to west
Sex	Female	**Age**	Early 20s
Pathologies	None		
Sample taken	Pollen sample, context 295, within coffin, 'gut' area		
Images			
Description	Dark brown soil sample, originally wet and set hard		
Storage history	In plastic bags	**Sampling procedure**	Crushed to homogenise, random sample, spatula
Comments			

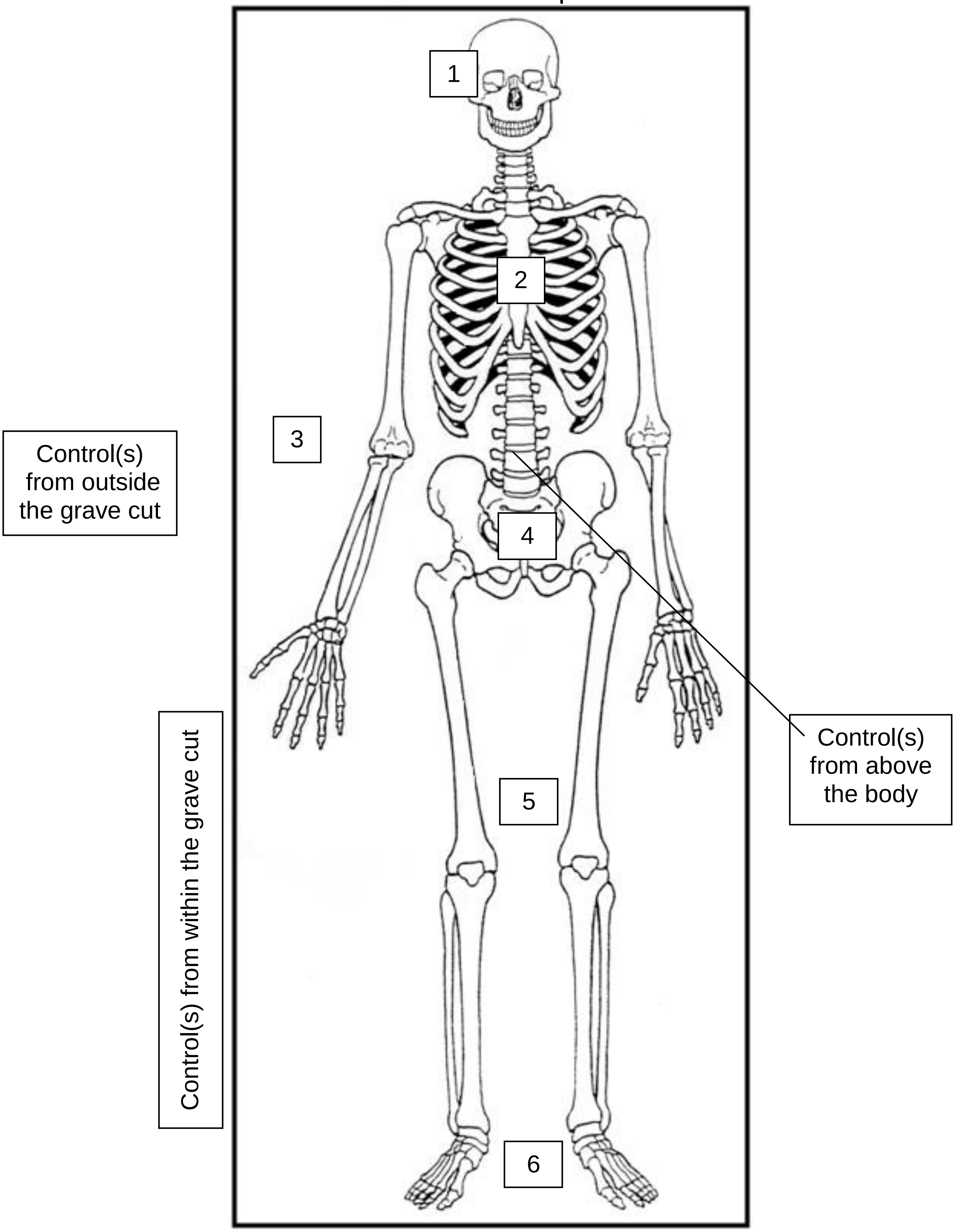
2.2 Ideal sample collection strategy



2: moderate soil/silt present



3: minimal soil/silt present



2.3 COSHH forms

Disc 1, Appendix 2, File 1

Solvent extraction, derivatisation and GC-MS analysis of modern resins and organic residues from Roman period inhumations

Disc 1, Appendix 2, File 2

Oxidation and epimerisation of oleanolic and ursolic acids and characterisation of the products

2.4 Standard operating procedures

Disc 1, Appendix 2, File 3

Solvent extraction of modern and archaeological natural resin samples: Standard Operating Procedure (SOP)

Disc 1, Appendix 2, File 4

Derivatisation of modern and archaeological natural resin samples: Standard Operating Procedure (SOP)

Disc 1, Appendix 2, File 5

GC-MS analysis and instrument maintenance (COSHH and SOP)

Appendix 3. Analysis of reference materials

Key compounds present in resins and gum-resins of interest (TMS derivatives). See **Table 6.1** for details of the modern reference materials analysed. Tables compiled by the author based on references given in the text and analysis of reference samples. Structures obtained from EMBL-EBI (2016) and NIST (2016).

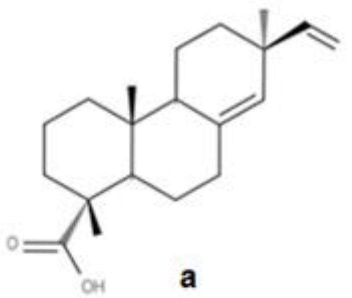
3.1 Pinaceae resins (*Abies* spp. *Cedrus* spp., *Larix* spp., *Picea* spp., *Pinus* spp.)

Name of compound	$M^{+\cdot}$	M-15	BP	Key fragment ions	Structure
Functionalised compounds					
Pimaric/*iso*pimaric acid isomers	374	359	241/73	257, 157, 143, 133, 105, 91	
Pimaric acid (13β-pimara-8,15-dien-18-oic acid)	374	359	73	257, 241, 157, 133, 91	**a**
Sandaracopimaric acid (13α-pimara-8,15-dien-18-oic acid)	374	359	121	257, 241; 143, 91	**b**
Palustric acid (abieta-8,13-dien-18-oic acid)	374	359	73	256, 241, 157, 143, 105, 91	**d**
*Iso*pimaric acid (pimara-7,15-dien-18-oic acid)	374	359	241	356, 241, 143, 105, 91	**c**
Didehydroabietic acid (abieta-6,8,11,13-tetraen-18-oic acid)	370	355	237	252, 195, 181, 153, 143	
Dehydroabietic acid (abieta-8,11,13-trien-18-oic acid)	372	357	239	255, 185, 173/1, 143, 129, 73	**f**
*Abietic acid (abieta-7,13-dien-18-oic acid)	374	359	256	257, 241, 213, 185, 105, 73	**e**
7-oxodehydroabietic acid (7-oxoabieta-8,11,13-trien-18-oic acid)	386	371	253	327, 268, 187, 143, 73	
Defunctionalised compounds					
Norabieta-8,11,13-trienes	256	241	241/238	243/2, 213, 199, 185,159, 117	
19-norabieta-4,8,11,13-tetraene (+ isomers)	254	239	239	195, 165, 141, 91	
Dehydroabietane (abieta-8,11,13-triene)	270	255	255	199, 185, 173, 159, 143, 129	
Abietane	272	257	257	241, 187, 161, 119, 105	
1,2,3,4-tetrahydroretene	238	223	223	224, 195, 181, 178/9, 165/166	**h**
Retene (isopropylmethylphenanthrene)	234	219	219	220, 205/4/3, 191, 189, 173, 83	
9-methylretene (isopropyldimethylphenanthrene)	248	233	233	218, 203, 189, 109, 101	
Abieta-8,11,13-trien-3-one	284	269	269	227, 213, 199, 185, 143, 115	**g**
Methyl dehydroabietate (methyl abieta-8,11,13-trien-18-oate)	314	299	239	299, 255, 239, 141, 129/8,	**i**
Methyl 7-oxodehydroabietate (methyl 7-oxoabieta-8,11,13-trien-18-oate)	328	253	253	269, 254, 213, 187	

*Only in Pinaceae resins

Larix spp.: additional labdane-skeleton diterpenic compounds, epimanool (labda-8,14-dien-13-ol) ($M^{+\cdot}$ 302; BP 137), larixol (labda-8,14-dien-6,13-diol) ($M^{+\cdot}$ 450; Fragments 153, 109, 69), larixyl acetate (6α-acetoxylabda-8,14-diene-13-ol) ($M^{+\cdot}$ 420), abieta-7,13-dien-18-ol ($M^{+\cdot}$ 360), labda-8,13-dien-15-oic acid ($M^{+\cdot}$ 376; BP 156), labda-8,13-dien-15,19-dioic acid ($M^{+\cdot}$ 478; BP 244). *Abies* spp.: *cis*-abienol (labda-12,14-dien-8-ol) ($M^{+\cdot}$ 362).

NB: Cupressaceae resins (*Juniperus* spp., *Cupressus* spp., *Tetraclinis* spp.) also contain many of these compounds but additionally include: ferruginol ($M^{+\cdot}$ 358; BP 358; Key fragments 310, 275, 261=247, 221, 205, 141, 115), totarol ($M^{+\cdot}$ 358; BP 343; Key fragments 315, 301, 287, 273, 261/0much<247, 231), sempervirol ($M^{+\cdot}$ 358; BP 343, Key fragments 315, 301, 287, 273, 261/0<247, 231), 3-hydroxyferruginol ($M^{+\cdot}$ 446; BP 446; Key fragments 431, 299, 259, 219).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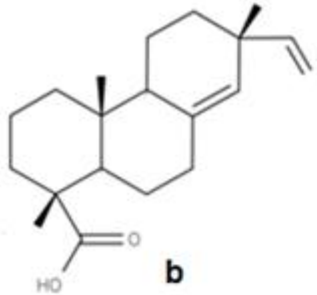
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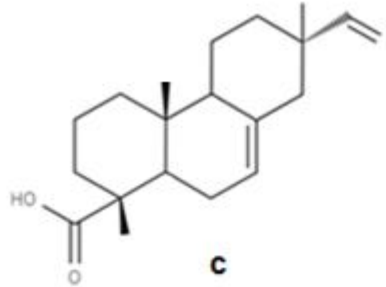
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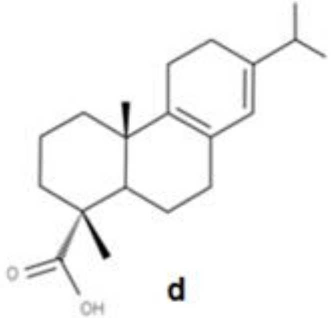
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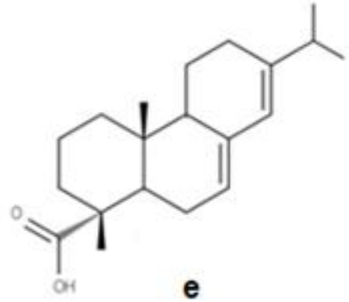
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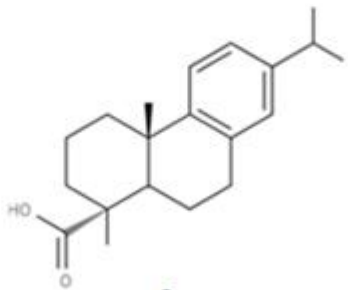
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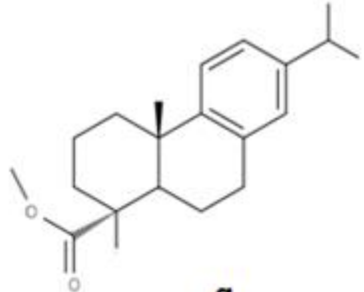
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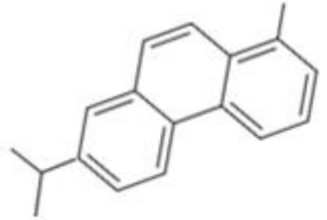
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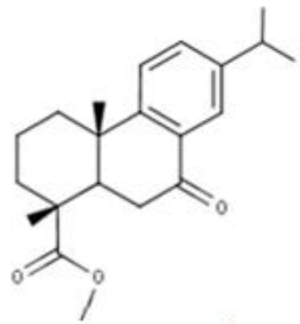
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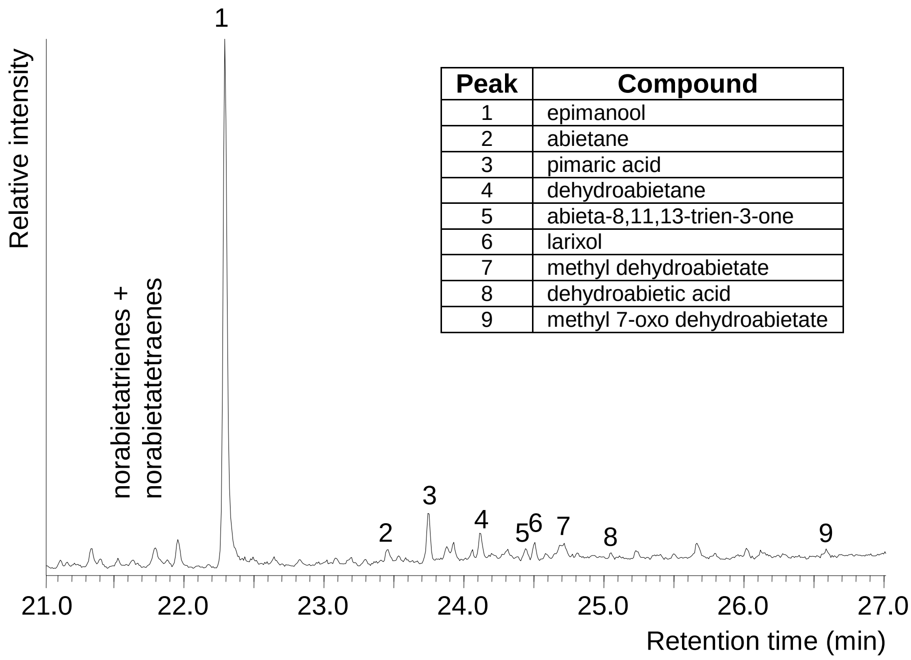
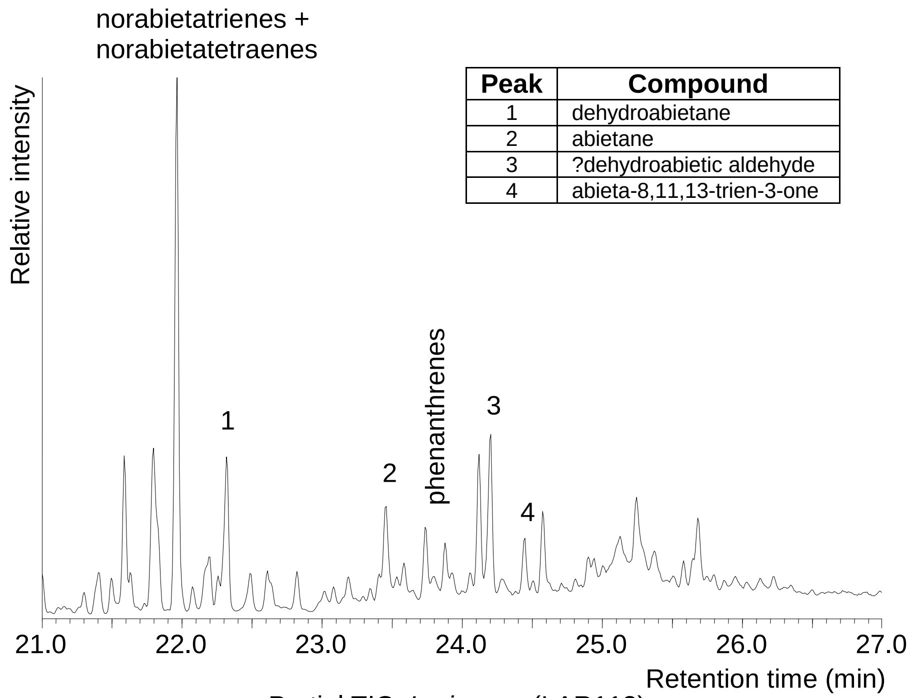


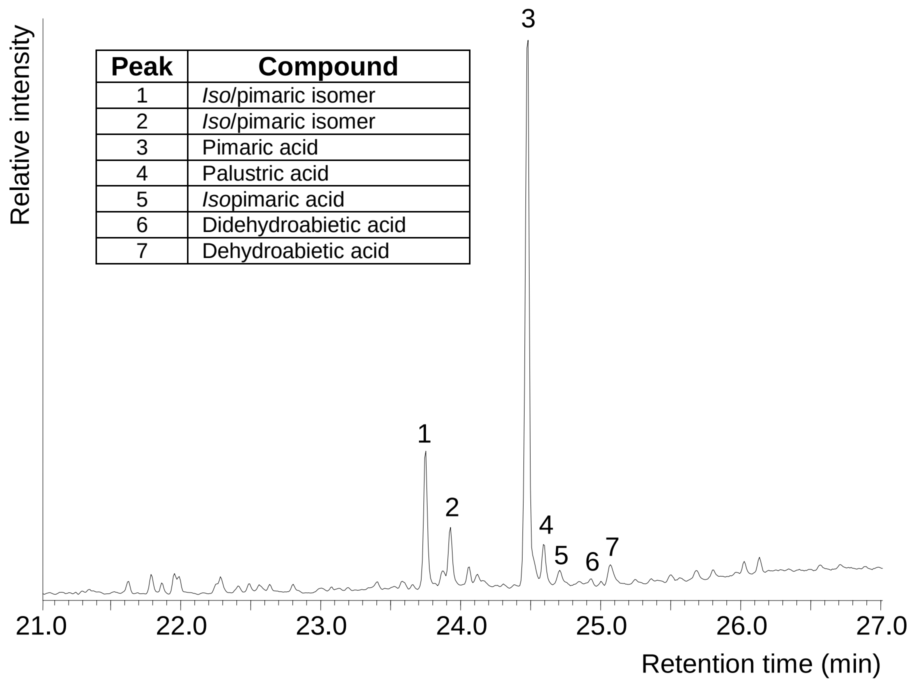
Peak	Compound
1	epimanool
2	abietane
3	pimaric acid
4	dehydroabietane
5	abieta-8,11,13-trien-3-one
6	larixol
7	methyl dehydroabietate
8	dehydroabietic acid
9	methyl 7-oxo dehydroabietate

Partial TIC: *Cedrus libani* (CED009)



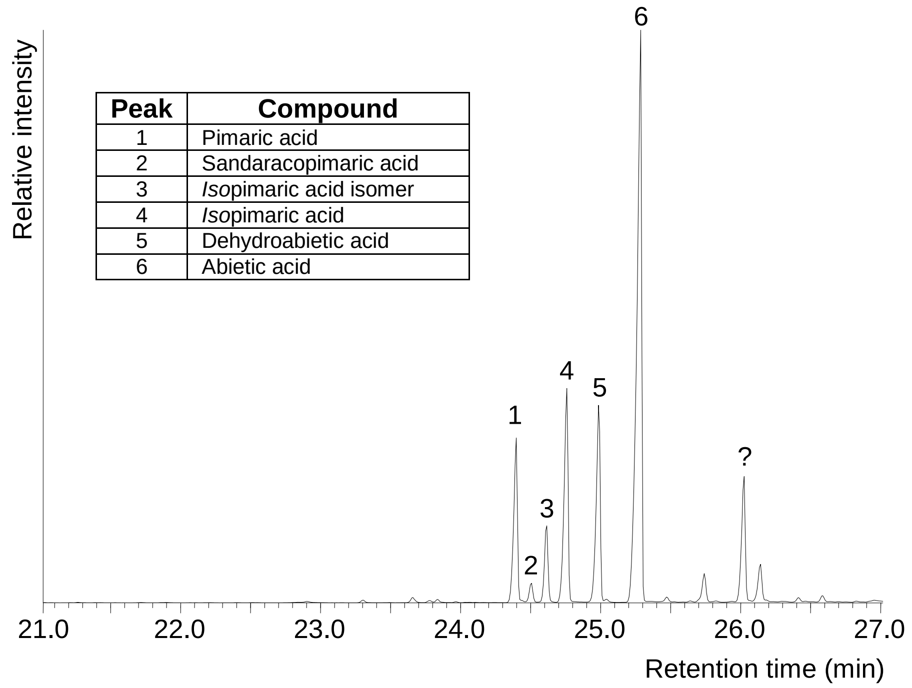
Peak	Compound
1	dehydroabietane
2	abietane
3	?dehydroabietic aldehyde
4	abieta-8,11,13-trien-3-one

Partial TIC: *Larix* spp. (LAR113)



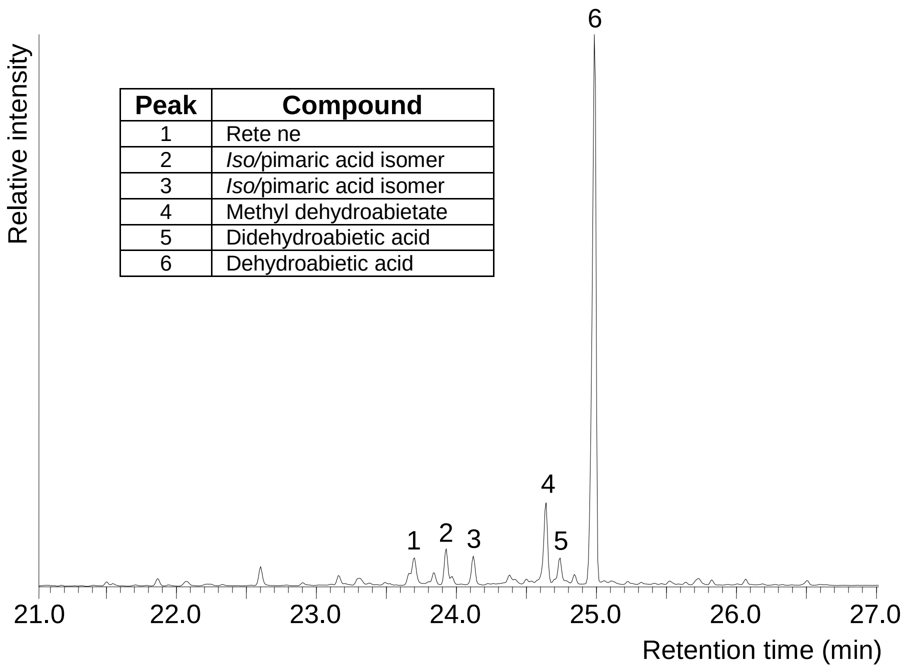
Peak	Compound
1	*Iso*/pimaric isomer
2	*Iso*/pimaric isomer
3	Pimaric acid
4	Palustric acid
5	*Iso*pimaric acid
6	Didehydroabietic acid
7	Dehydroabietic acid

Partial TIC: *Pinus pinaster* (PIN065)



Peak	Compound
1	Pimaric acid
2	Sandaracopimaric acid
3	*Iso*pimaric acid isomer
4	*Iso*pimaric acid
5	Dehydroabietic acid
6	Abietic acid

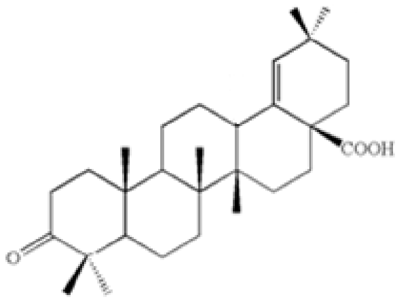
Partial TIC: *Pinus sylvestris* (NRKFZ)



Partial TIC: *Pinus sylvestris* tar (PST121)

3.2 *Pistacia* spp. resins

Name of compound	$M^{+\cdot}$	M-15	BP	Key fragment ions	Structure
tirucallol	426	411	411	393, 327, 281, 241, 207, 189, 135, 121	
?olean-en-one isomer	424	409	409	383, 311, 271, 257, 231, 205, 187, 133, 121	
?norolean-dien-one isomer	408	393	393	311, 241, 229, 215, 187, 173, 129, 109	
?olean-en-one isomer	424	409	409	393, 355, 271, 257, 245, 231, 205, 187, 121	
28-norolean-12-en-3-one	410	395	204	381, 313, 245, 215, 189, 175, 133	
β-amyrenone (olean-12-en-3-one)	424	409	218	218, *203 much>189,* 122, 95, 55	
β-amyrin (3β-hydroxy-olean-12-en-3-ol)	498	483	218	483, 409/7, 393, 279, *203 much>189*, 190, 122	
olean-18-en-3-one	424	409	93	408, 393, 355, 313, 245, 205, 189, 163, 133	
28-norolean-17-en-3-one	410	395	163	393, 279, 257, 218, 203, 133, 119	
28-norolean-12,17-dien-3-one	408	393	408	379, 339, 258, 229, 216, 203, 189, 173, 129	
?olean-dien-3-one	422	407	422	311, 216, 203, 189, 175, 161, 145, 131	
?olean-dien-3β-ol	496	481	409	391, 218, 203, 189, 175, 147, 133	
moronic acid (3-oxoolean-18-en-28-oic acid)	526	511	189	409, 391, 320, 307, 219, 203, 133, 119	**a**
oleanonic acid (3-oxoolean-12-en-28-oic acid)	526	511	203	408, 393, 320, 307, 219, 189, 133, 119	**b**
oleanolic acid (3β-hydroxyolean-12-en-28-oic acid)	600	585	203	511, 232, 189	
oleanonic aldehyde (3-oxoolean-12-en-28-al)	438	423	203	409, 320, 232, 189, 175, 133, 119, 105	
?a triterpen-ol-one (similar to betulone but with oleanane-skeleton?)	512	497	73	407, 422, 271, 257, 245, 203, 187, 143, 109	
?a triterpen-diol (similar to betulin but with oleanane-skeleton?)	586	571	73	512, 497, 407, 391, 311, 257, 241, 187, 143	
*iso*masticadienonic acid (3-oxotirucalla-8,24-dien-26-oic acid)	526	511	511	421, 393, 307, 257, 243, 213, 185, 169, 119	**d**
masticadienonic acid (3-oxotirucalla-7,24-dien-26-oic acid)	526	511	511	421, 393, 311, 257, 213, 185, 169, 143, 119	**c**
3α-acetoxy-3-epii*so*masticadienolic acid (3α-acetoxy-3-epioxotirucalla-8,24-dien-26-oic acid)	570	555	495	511, 423, 393, 241, 213, 189, 169, 133	
? *iso*masticadienolic aldehyde (3-oxotirucalla-8,24-dien-26-al)	440	425	409	423, 315, 281, 245, 203=189, 175, 133	
3α-acetoxy-3-epimasticadienolic acid (3α-acetoxy-3-epioxotirucalla-7,24-dien-26-oic acid)	570	555	495	511, 423, 405, 241, 213, 185, 169, 133	



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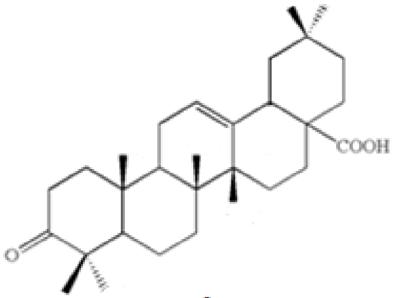

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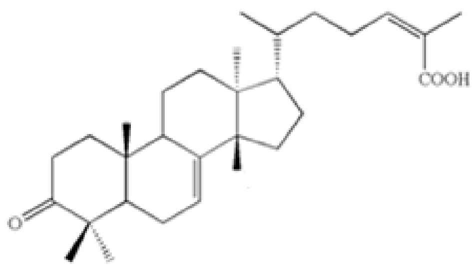

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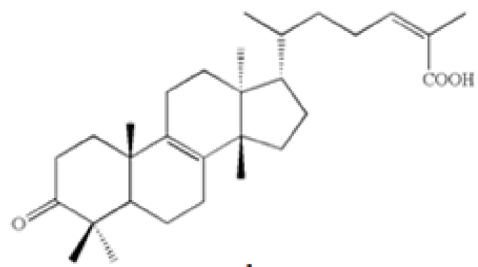

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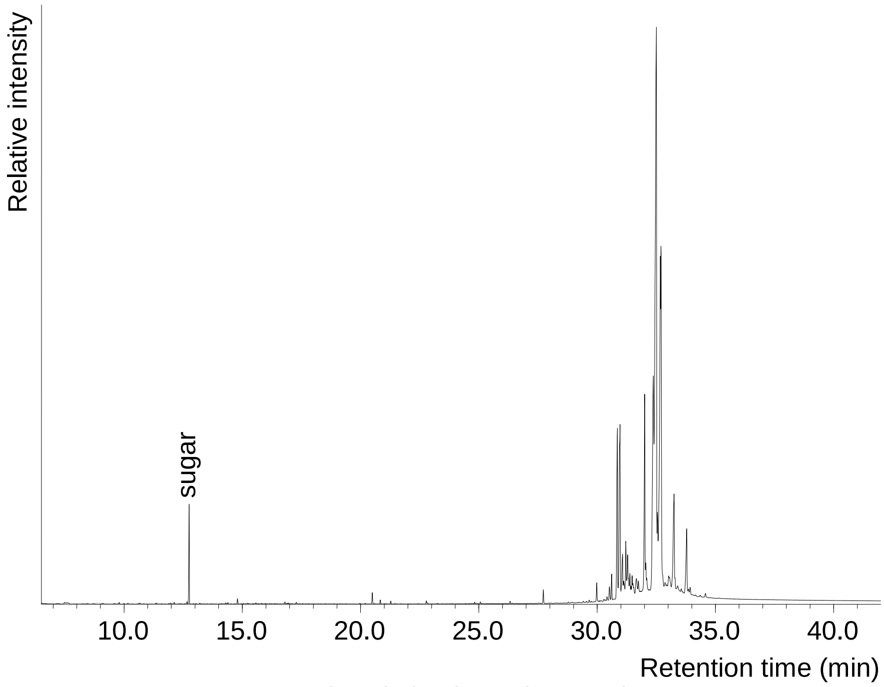
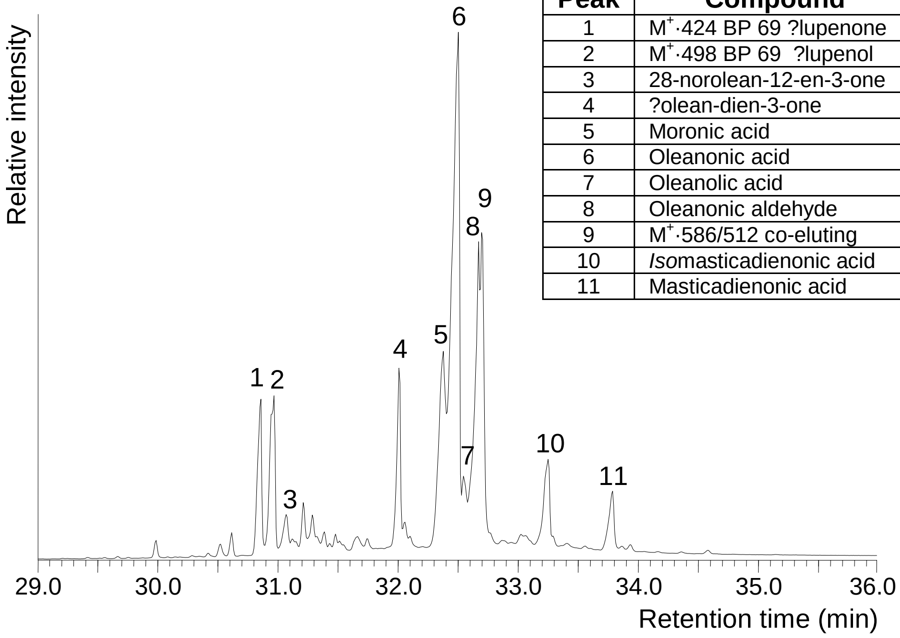
NB: the shift in retention time occurred after essential maintenance by Agilent which required replacement of the ion source

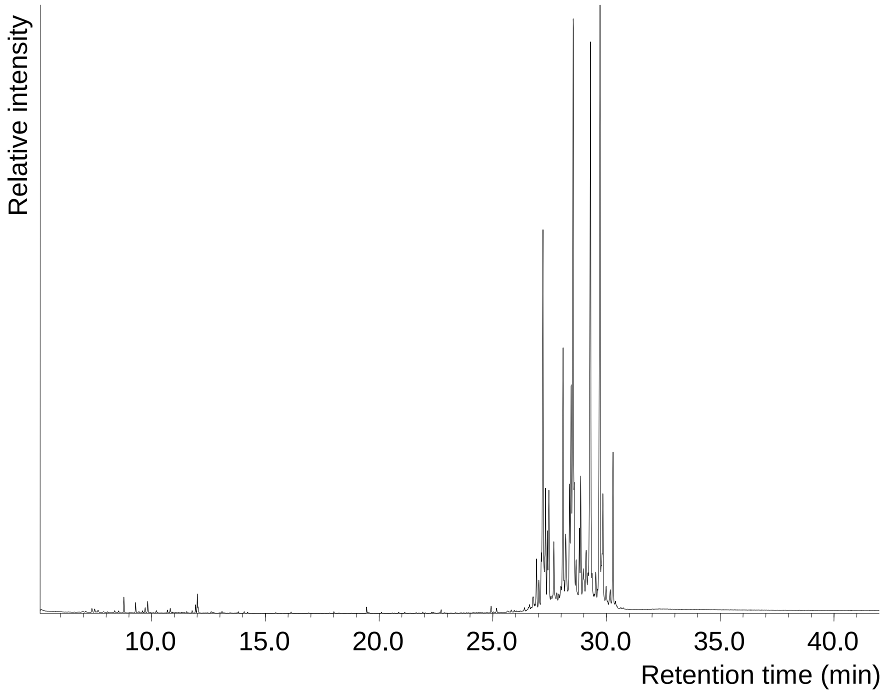


TIC: *Pistacia lentiscus* (PLT012)

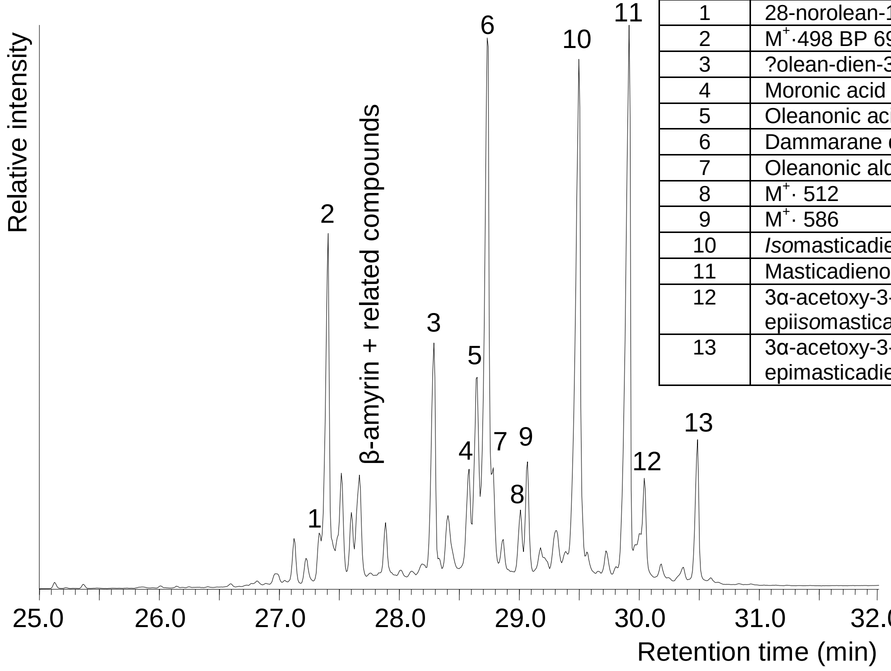


Peak	Compound
1	$M^{+}\cdot$424 BP 69 ?lupenone
2	$M^{+}\cdot$498 BP 69 ?lupenol
3	28-norolean-12-en-3-one
4	?olean-dien-3-one
5	Moronic acid
6	Oleanonic acid
7	Oleanolic acid
8	Oleanonic aldehyde
9	$M^{+}\cdot$586/512 co-eluting
10	*Iso*masticadienonic acid
11	Masticadienonic acid

Partial TIC: *Pistacia lentiscus* (PLT012)

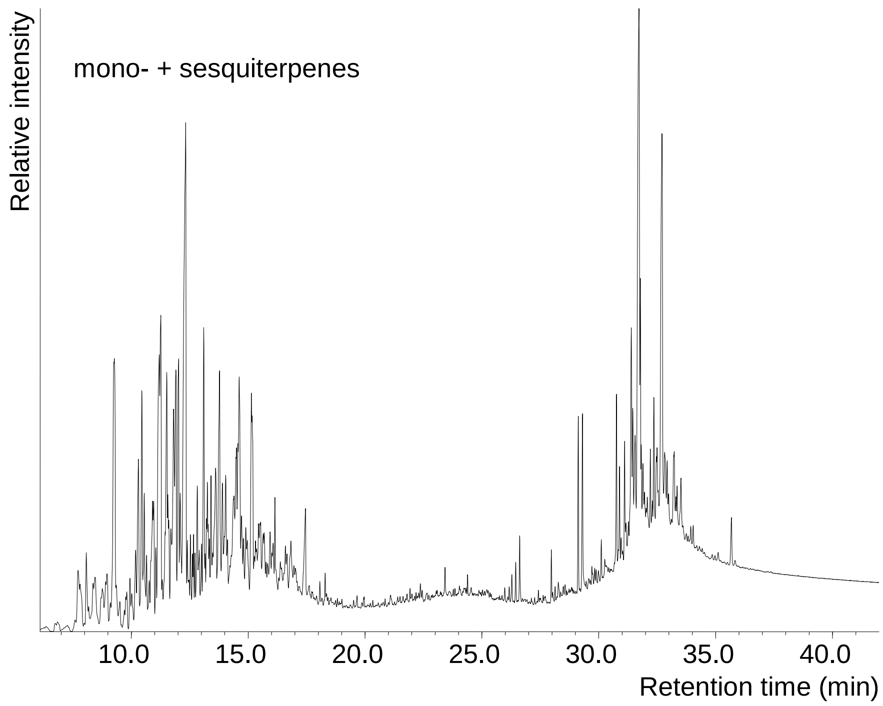


TIC: *Pistacia lentiscus* (PA)

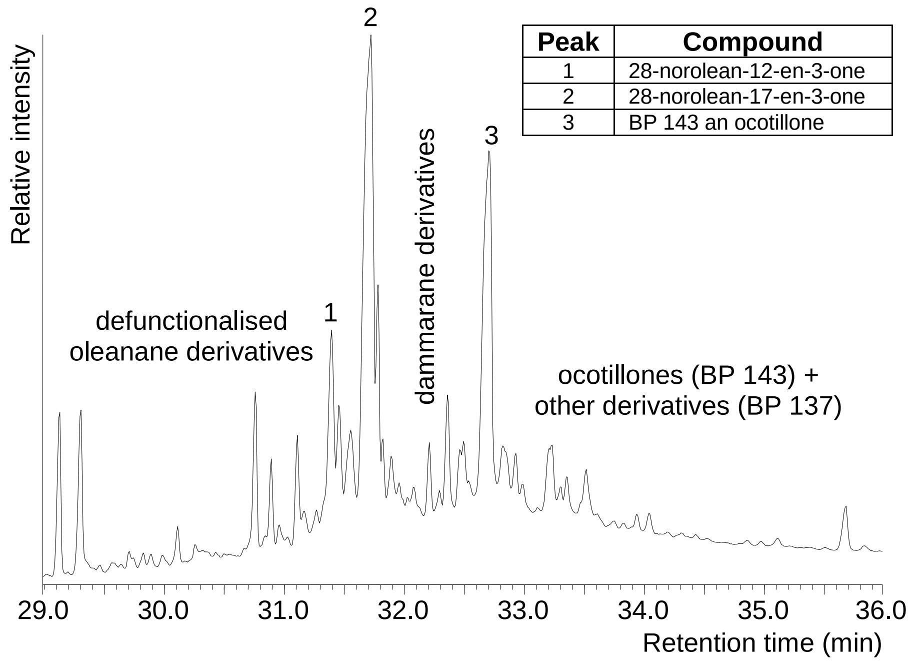


Peak	Compound
1	28-norolean-12-en-3-one
2	M^{+}·498 BP 69 ?lupenol
3	?olean-dien-3-one
4	Moronic acid
5	Oleanonic acid
6	Dammarane derivative
7	Oleanonic aldehyde
8	M^{+}· 512
9	M^{+}· 586
10	*Iso*masticadienonic acid
11	Masticadienonic acid
12	3α-acetoxy-3-epi*iso*masticadienolic acid
13	3α-acetoxy-3-epimasticadienolic acid

Partial TIC: *Pistacia lentiscus* (PA)

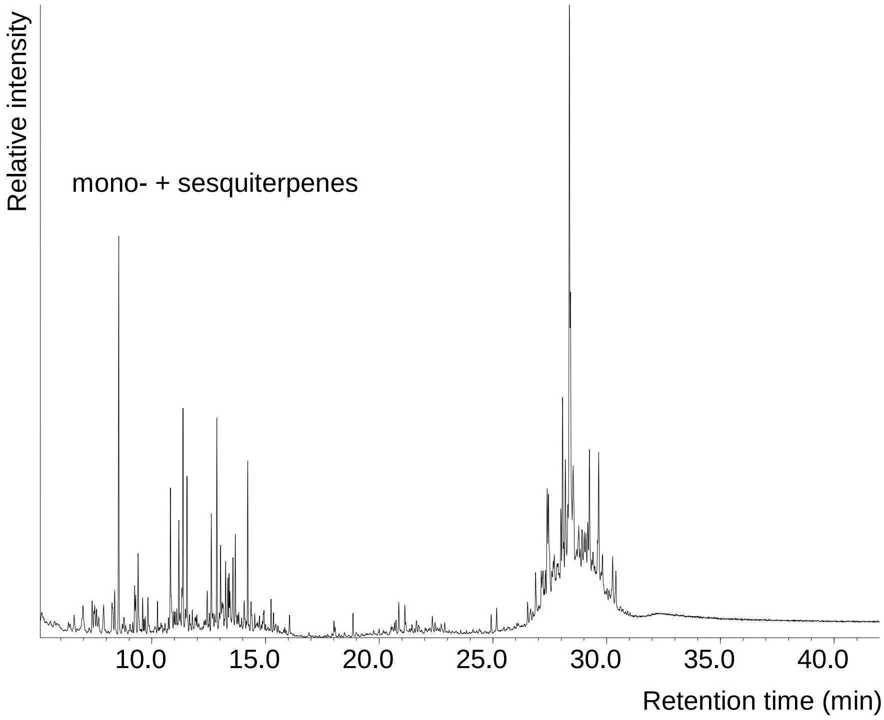


TIC: *Pistacia terebinthus* (PTC055)

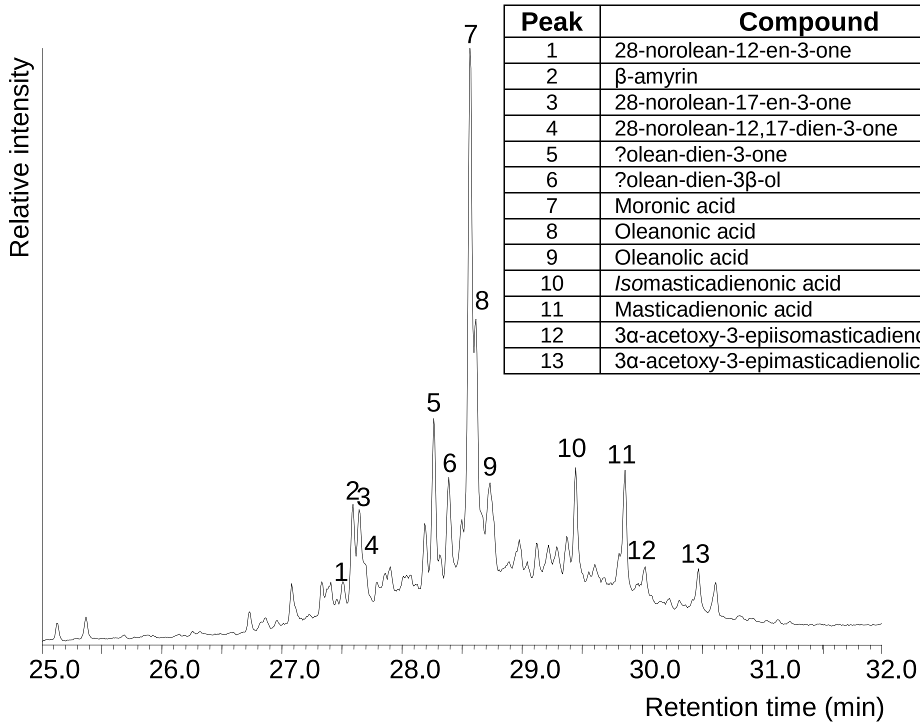


Peak	Compound
1	28-norolean-12-en-3-one
2	28-norolean-17-en-3-one
3	BP 143 an ocotillone

Partial TIC: *Pistacia terebinthus* (PTC055)

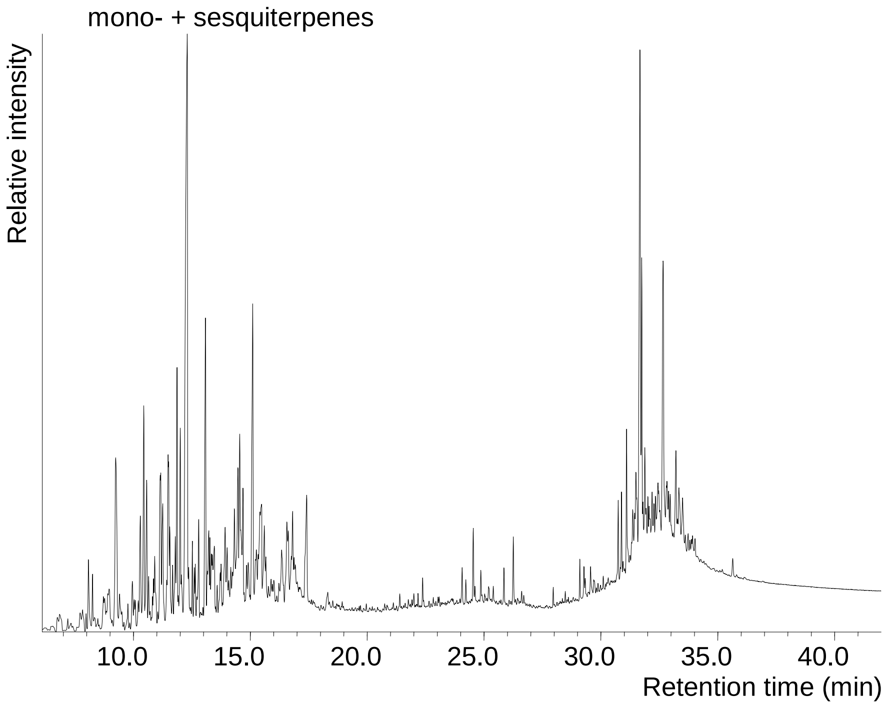


TIC: *Pistacia terebinthus* (PTP056)

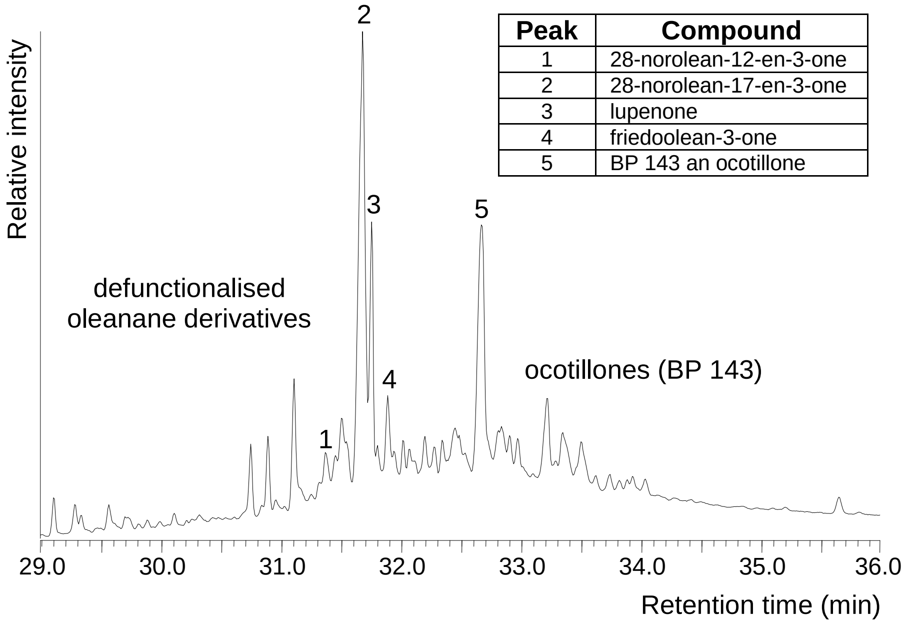


Peak	Compound
1	28-norolean-12-en-3-one
2	β-amyrin
3	28-norolean-17-en-3-one
4	28-norolean-12,17-dien-3-one
5	?olean-dien-3-one
6	?olean-dien-3β-ol
7	Moronic acid
8	Oleanonic acid
9	Oleanolic acid
10	*Iso*masticadienonic acid
11	Masticadienonic acid
12	3α-acetoxy-3-epi*iso*masticadienolic acid
13	3α-acetoxy-3-epimasticadienolic acid

Partial TIC: *Pistacia terebinthus* (PTP056)



TIC: *Pistacia khinjuk* (PKI035)



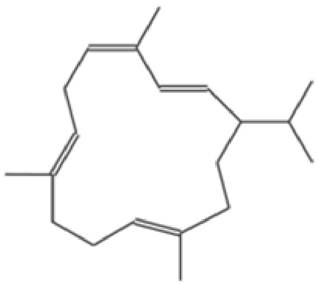
Peak	Compound
1	28-norolean-12-en-3-one
2	28-norolean-17-en-3-one
3	lupenone
4	friedoolean-3-one
5	BP 143 an ocotillone

Partial TIC: *Pistacia khinjuk* (PKI035)

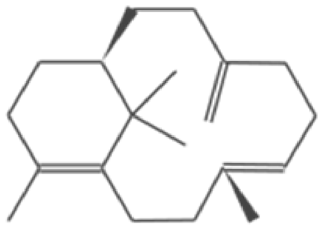
NB: mono- and sesquiterpenes in *P. lentiscus* are far more limited in number and in lower abundance in comparison with their range and abundance in *P. terebinthus* and *P. khinjuk*; the absence of the classic quartet of triterpenoids in examples of latter species' is also worthy of note and may have significant implications for their identification in the archaeological record in comparison with *P. lentiscus*.

3.3 *Boswellia* spp. gum-resins

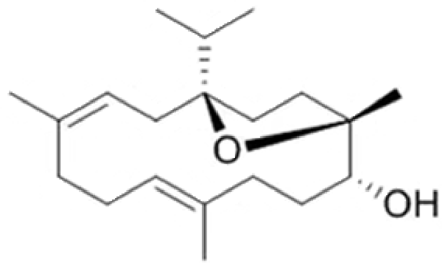
Name of compound	$M^{+\cdot}$	M-15	BP	Key fragment ions	Structure
Diterpenic compounds					
Cembrene A (1,5,9-trimethyl-12(propan-2-yl)cyclotetradecatriene)	272	257	?	243, 229, 189, 161, 119, 93	
Cembrene C (1,7,11-trimethyl-4(propanyl)cyclotetradecatetraene)	272	257	272	243, 229, 189, 161, 119, 93	**a**
Verticilla-4(20),7,11-triene	272	257	257	229, 189, 161, 133, 121, 93	**b**
Incensol (1,5,9-trimethyl-12-propan-2-yl-15-oxadicyclopentadeca-5-9-dien-2-ol)	306	291			**c**
Incensol (TMS derivative)	378	363	43	263, 156, 125, 107, 71	
Incensol acetate (1,5,9-trimethyl-12-propan-2-yl-15-oxabicyclopentadeca-5,9-dien-2-yl acetate)	348	333	43	305, 150, 125, 71	
Incensol oxide (1-*iso*propyl-5,9,13-trimethyl-4,16-dioxatricyclohexadec-8-en-12-ol) (underivatised)	322	307	43	209, 109, 71	
Incensol oxide acetate (1-*iso*propyl-5,9,13-trimethyl-4,16-dioxatricyclohexadec-8-en-12-yl acetate)	364			209, 125, 107, 43	
m-camphorene in *B. serrata*	272		69	257, 229, 203, 187, 161, 133, 119, 91	
p-camphorene in *B. serrata*	272		69	257, 229, 203, 187, 161, 133, 119, **93**	
Isomers of α-phellandrene dimers in B. frereana	272		93	136, 121, 105, 77 (virtually nothing else)	



a

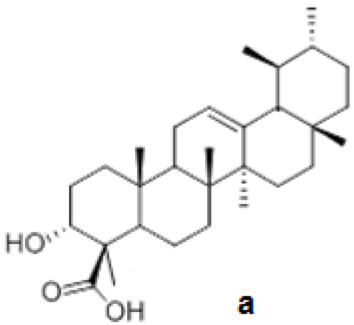


b



c

Name of compound	$M^{+\cdot}$	M-15	BP	Key fragment ions	Structure
Triterpenic compounds					
24-norolean-3,9(11),12-triene	392	377	392	281, 271, 239, 218, 203, 185, 133, 119	
24-norursa-3,9(11),12-triene	392	377	392	281, 267, 239, 213, 203, 185, 133, 119	
24-norolean-3,12-diene	394	379	203	218, 189, 175	
24-norursa-3,12-diene	394	379	218	203, 189, 175	
24-norlupa-3,12-diene	394	379	175	218, 203, 189	
3α-hydroxy-olean-12-en-3-ol (3-epi-β-amyrin)	498	483	203	408, 218, 189	
3α-hydroxy-urs-12-en-3-ol (3-epi-α-amyrin)	498	483	218	408, 203. 189	
3α-lup-20(29)-en-3-ol (3-epi-lupeol)	498	483	189	408, 218, 203, 175	
olean-12-en-3-one (β-amyrenone)	424	409	218	218, *203 much>189,* 122, 95, 55	
3β-hydroxy-olean-12-en-3-ol (β-amyrin)	498	483	218	409/7, 393, 279, *203 much>189*, 190, 122, 95, 73	
24-norursa-3,12-dien-11-one	408	393	232	353, 273, 255, 161, 135	
urs-12-en-3-one (α-amyrenone)	424	409	218	313, *203 c.=189,* 133, 122, 55	
3β-hydroxy-urs-12-en-3-ol (α-amyrin)	498	483	218	465, 408, 393, 279, 207, *203 c.=189,* 135, 122	
lup-20(29)-en-3-one (lupenone)	424	409	205/95	313, 218, 203, 189, 109, 55	
3β-lup-20(29)-en-3-ol (lupeol)	498	483	189	408, 393, 279, 218, 203, 175, 135, 121, 109, 73	
3α-hydroxy-olean-12-en-24-oic acid (α-boswellic acid)	600	585	218	510, 495, 382, 292, 281, *203>189*, 161, 107	**b**
3α-hydroxy-urs-12-en-24-oic acid (β-boswellic acid)	600	585	218	510, 495, 382, 292, *203 c.= 189*, 161, 133, 107	**a**
3α-hydroxy-lup-20(29)-en-24-oic acid	600	585	472	510, 292, 218, 203, 191, 189, 175, 161, 147/9, 121	
3α-*O*-acetyl-olean-12-en-24-oic acid (3-*O*-acetyl-α-boswellic acid)	570	555	203	510, 495, 393, 352, 292, 218, *much>189*, 161, 105	**d**
3α-*O*-acetyl-urs-12-en-24-oic acid (3-*O*-acetyl-β-boswellic acid)	570	555	218	510, 495, 393, 352, 292, *203 c.=189,* 161, 133, 119	**c**
3α-*O*-acetyl-lup-20(29)-en-24-oic acid	570	555	73	510, 292, 173	
3-oxo-8,9,24,25-tetradehydro-tirucallic acid	526	511	73	444, 393, 359, 354, 297, 189, 119	
3-hydroxy-8,9,24,25-tetradehydro-tirucallic acid	600	585	495/73	377, 310, 295, 281, 189, 129	
3-*O*-acetyl-8,9,24,25-tetradehydro-tirucallic acid	570	555	423	495, 377, 281, 187, 133, 73	



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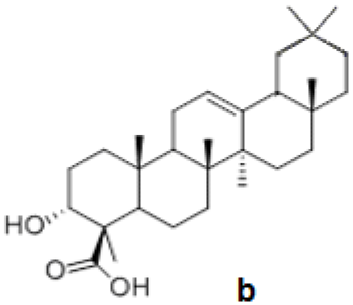

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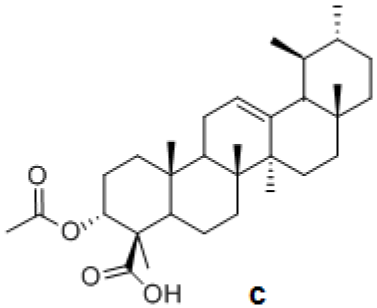

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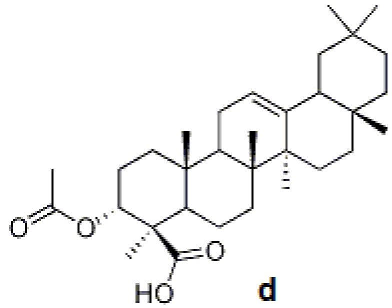

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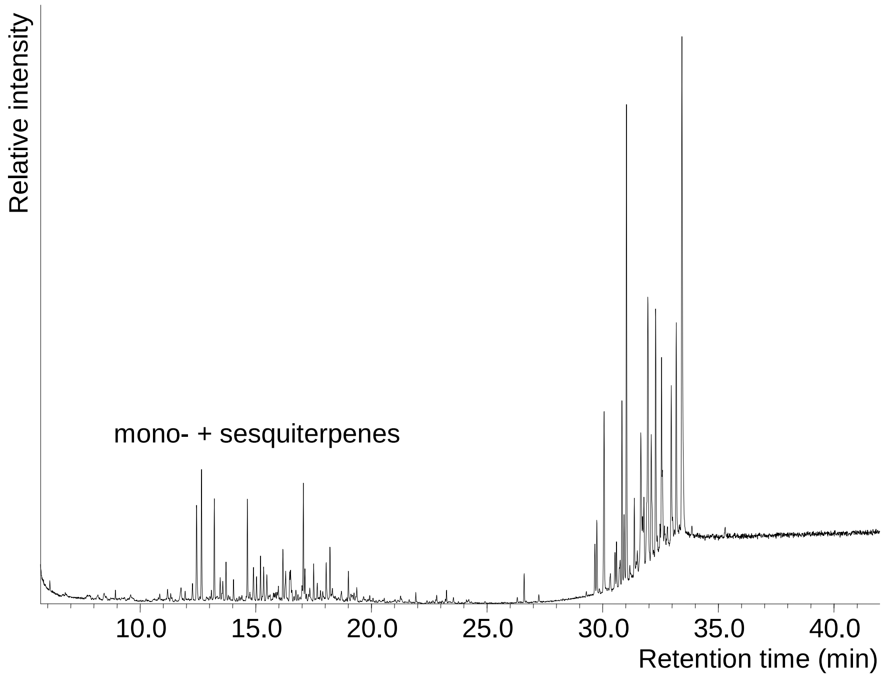
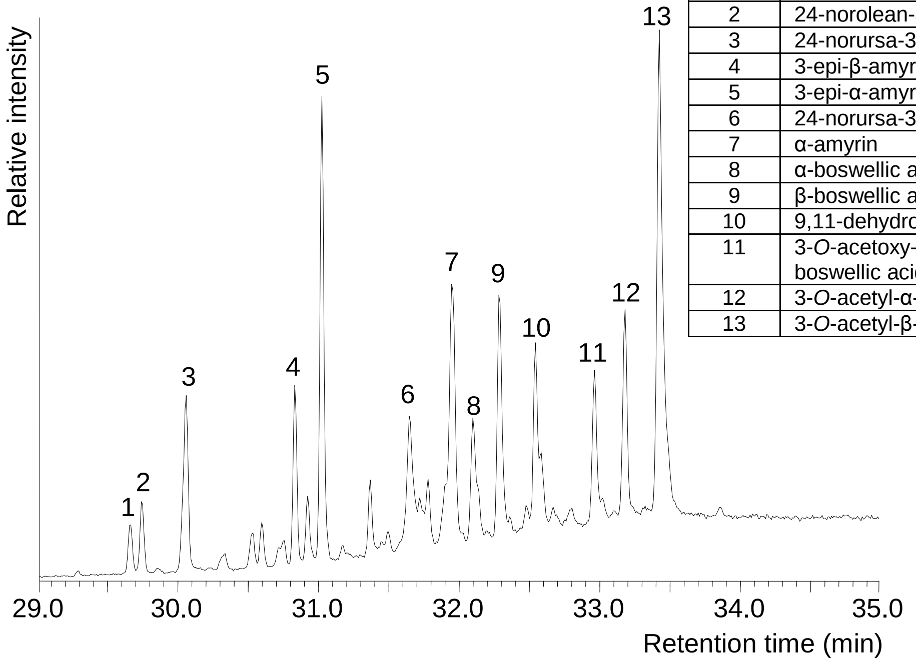
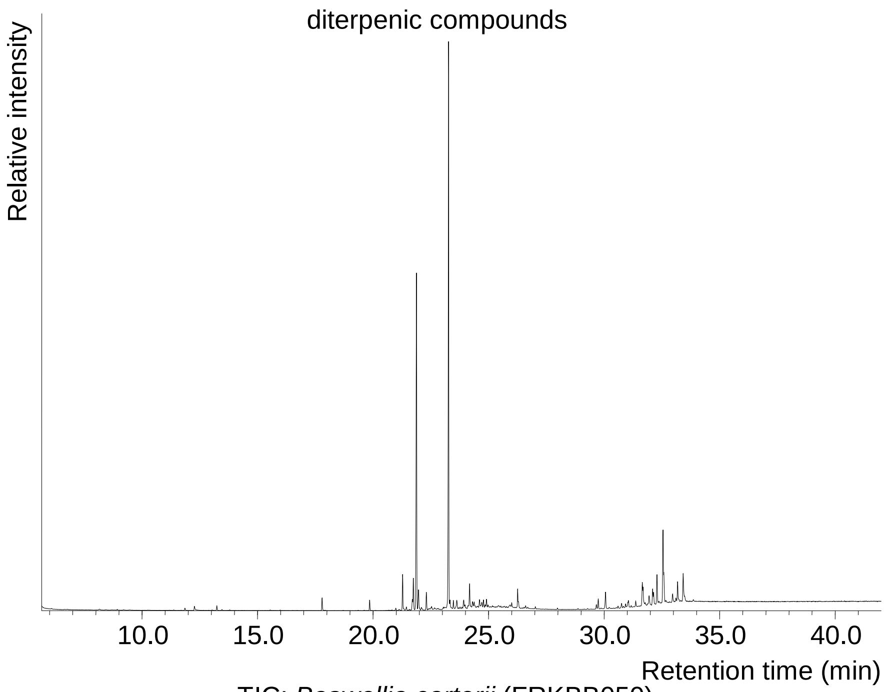


TIC: *Boswellia sacra* (FRKBB052)



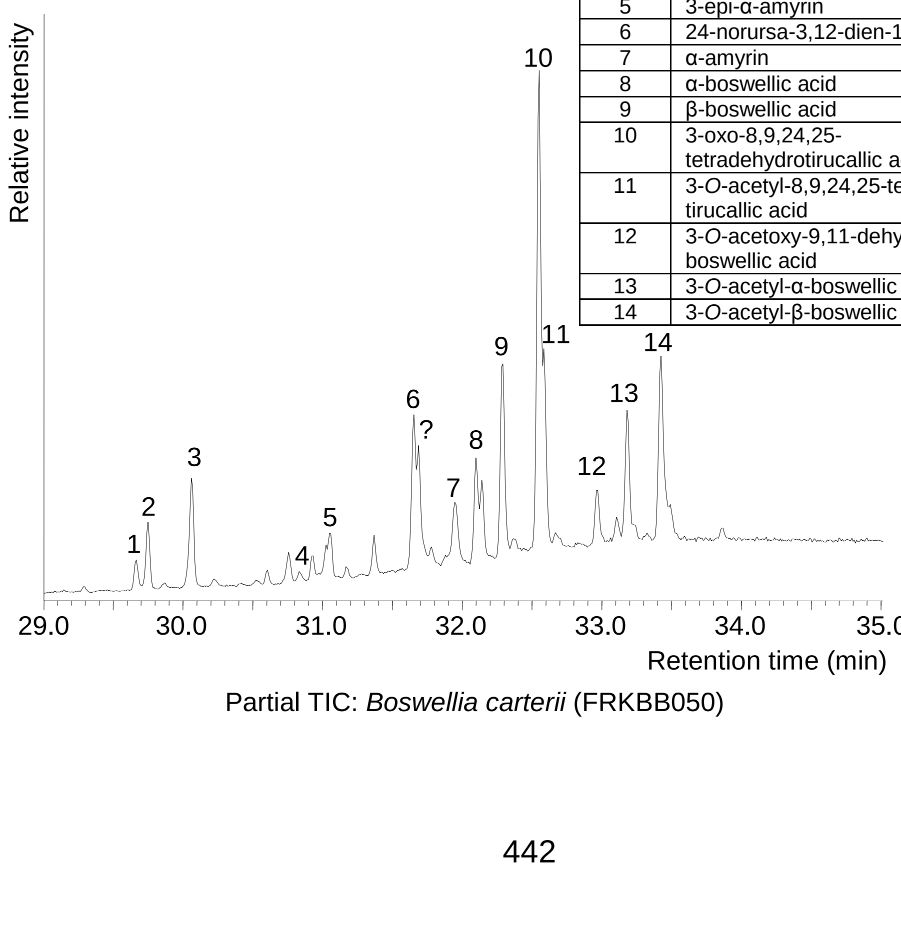
Peak	Name of compound
1	24-norursa-3,9(11),12-triene
2	24-norolean-3,12-diene
3	24-norursa-3,12-diene
4	3-epi-β-amyrin
5	3-epi-α-amyrin
6	24-norursa-3,12-dien-11-one
7	α-amyrin
8	α-boswellic acid
9	β-boswellic acid
10	9,11-dehydro-β-boswellic acid
11	3-*O*-acetoxy-9,11-dehydro-β-boswellic acid
12	3-*O*-acetyl-α-boswellic acid
13	3-*O*-acetyl-β-boswellic acid

TIC: *Boswellia sacra* (FRKBB052)

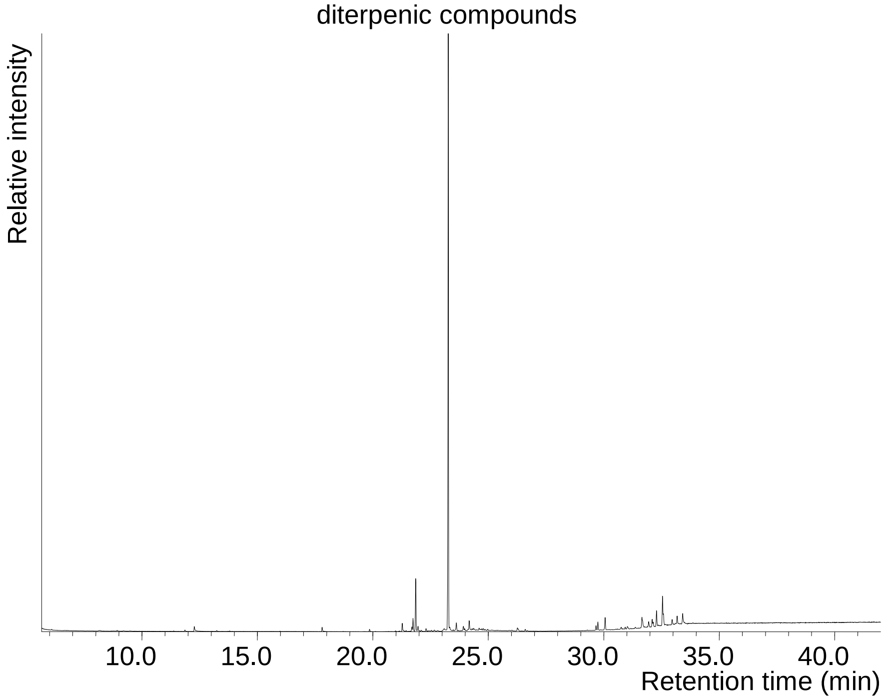


TIC: *Boswellia carterii* (FRKBB050)

Peak	Name of compound
1	24-norursa-3,9(11),12-triene
2	24-norolean-3,12-diene
3	24-norursa-3,12-diene
4	3-epi-β-amyrin
5	3-epi-α-amyrin
6	24-norursa-3,12-dien-11-one
7	α-amyrin
8	α-boswellic acid
9	β-boswellic acid
10	3-oxo-8,9,24,25-tetradehydrotirucallic acid
11	3-*O*-acetyl-8,9,24,25-tetradehydro-tirucallic acid
12	3-*O*-acetoxy-9,11-dehydro-β-boswellic acid
13	3-*O*-acetyl-α-boswellic acid
14	3-*O*-acetyl-β-boswellic acid

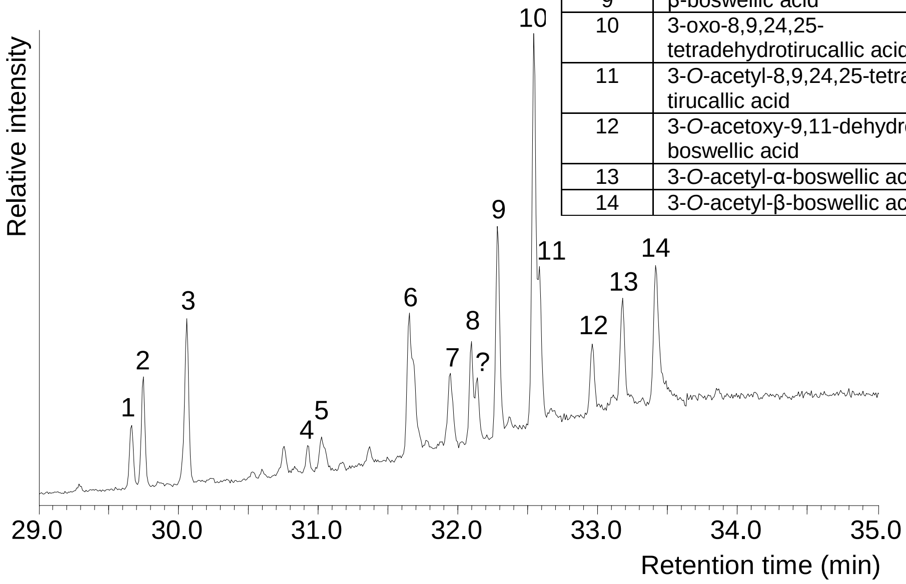


Partial TIC: *Boswellia carterii* (FRKBB050)

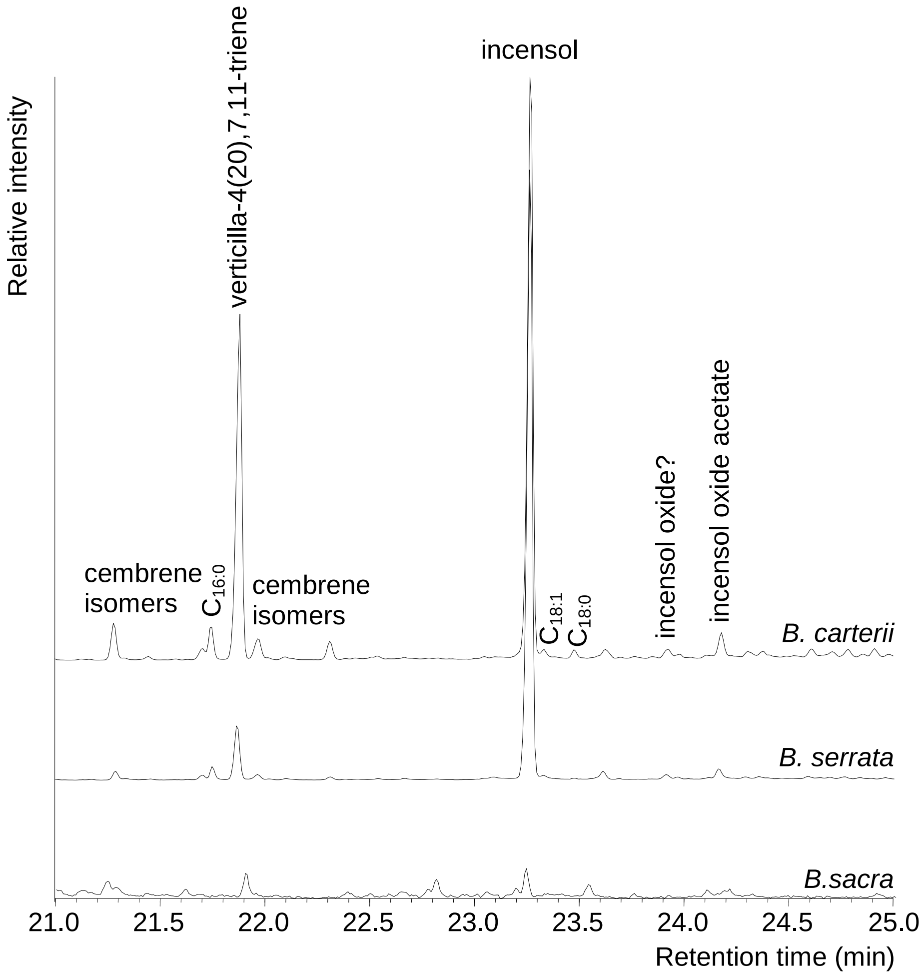


TIC: *Boswellia serrata* (FRKBB009)

Peak	Name of compound
1	24-norursa-3,9(11),12-triene
2	24-norolean-3,12-diene
3	24-norursa-3,12-diene
4	3-epi-β-amyrin
5	3-epi-α-amyrin
6	24-norursa-3,12-dien-11-one
7	α-amyrin
8	α-boswellic acid
9	β-boswellic acid
10	3-oxo-8,9,24,25-tetradehydrotirucallic acid
11	3-*O*-acetyl-8,9,24,25-tetradehydro-tirucallic acid
12	3-*O*-acetoxy-9,11-dehydro-β-boswellic acid
13	3-*O*-acetyl-α-boswellic acid
14	3-*O*-acetyl-β-boswellic acid



Partial TIC: *Boswellia serrata* (FRKBB009)



Partial TIC: diterpenic compounds, *Boswellia* spp. reference materials

3.4 Balsamic extracts: *Liquidambar orientalis* (storax) and *Styrax officinalis* (styrax)

Styrax officinalis: mainly contains free cinnamic and benzoic acids and corresponding esters with cinnamyl, p-coumaryl and coniferyl alcohols – very variable depending on species, collection method, growing conditions... (Hovaneissian *et al.* 2008; Pastorova *et al.* 1997)

$M^{+\bullet}$	BP	Key fragments	Name of compound
194	105	179, 135, 89, 77	benzoic acid
194	179	161, 151, 135, 73	4-hydroxybenzaldehyde
254	239	223, 195, 133, 112, 73	resorcinol (benzene-1,3-diol) x2 TMS
224	194	209, 165, 137, 73	vanillin (4-hydroxy-3-methoxy-benzaldehyde)
220	205	192, 161, 145, 131, 103, 75, 73	cinnamic acid (3-phenyl-2-propanoic acid)
282	267	223, 193, 126, 91, 73	3-hydroxybenzoic acid
282	73	267, 223, 193, 126, 91	4-hydroxybenzoic acid
312	297	282, 267, 253, 223, 193, 126, 73	vanillic acid (4-hydroxy-3-methoxy-benzoic acid)
206	117	191, 135, 91, 73	cinnamyl alcohol (3-phenylprop-2-en-1-ol)

Also look for:

- 1,4-benzenedicarboxylic acid TMS = M+ 310; bp 295; frags. 251, 221, 193, 178, 140, 103, 73
- 3,4-hydroxybenzoic acid TMS = M+ 370; bp 193; frags. 355, 311, 281, 267, 223, 165, 137, 73
- 2-methyl benzoic acid TMS = M+ 208; bp 193; frags. 149, 119, 91, 73

Liquidambar orientalis: main components are cinnamyl cinnamate, 3-phenylproanyl cinnamate, corresponding free alcohols, free benzoic and cinnamic acids; several triterpenoid acids with oleanane-skeletons, oleanolic, oleanonic and *epi*-oleanolic acids (Hafizoğlu 1982, *et al.* 1996; Modugno *et al.* 2006a; Pastorova *et al.* 1998).

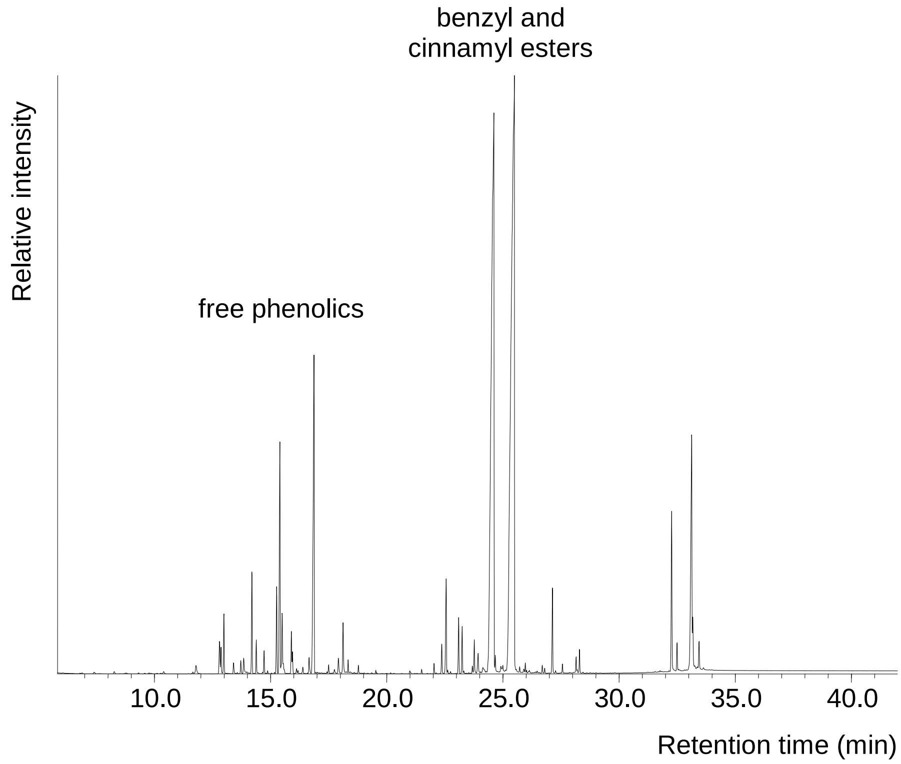
$M^{+\bullet}$	BP	Key fragments	Name of compound
194	105	179, 135, 89, 77	benzoic acid
254	239	223, 195, 133, 112, 73	resorcinol
220	205	192, 161, 145, 131, 103, 75, 73	cinnamic acid
206	117	191, 135, 91, 73	cinnamyl alcohol
296	206	281, 191, 179, 133, 89, 73	4-hydroxybenzenepropanol
308	73	293, 249, 219	p-hydroxycinnamic acid
600	203	585, 510, 482, 392/3, 320, 279, 189, 133	oleanolic acid
526	203	511, 393, 320, 306, 189, 173, 133	oleanonic acid
208	118	193, 117, 91, 89, 75, 73	3-phenyl-2-propanol

Huneck (1963): *Liquidambar orientalis*

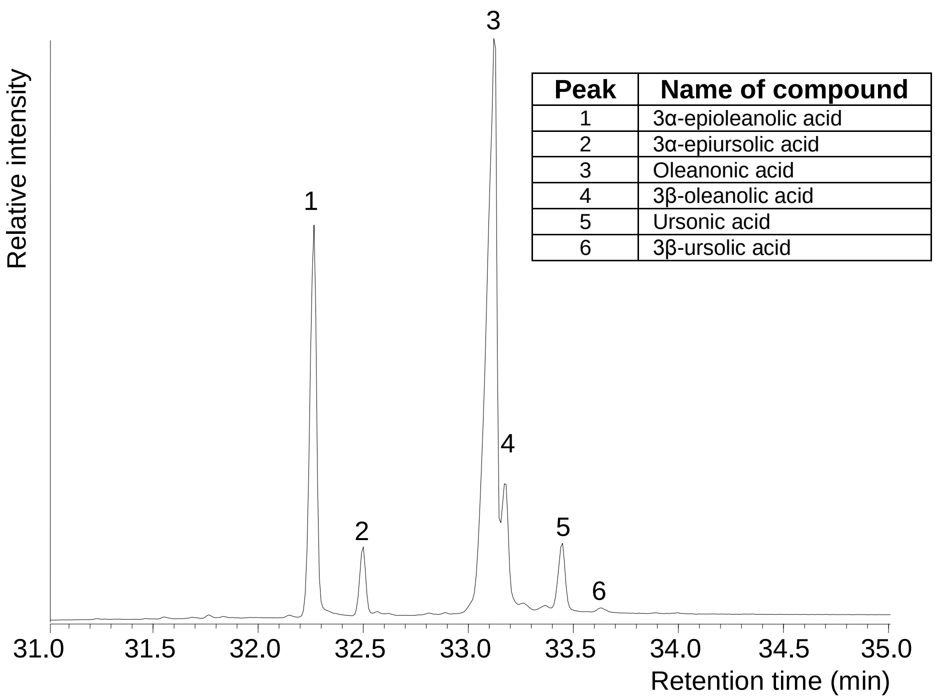
- 3-epi-oleanolic acid (Mr = 456; ME = 470;TMS = 600)
- oleanonic acid (Mr = 454; ME = 468;TMS = 526)
- ethyl esters = 484 and 482
- actyl-3-epi-oleanolic acid = 498
- acetyl MEs = 512 and 526 (TMS?)
- 3-epi-erythrodiol = 442 (TMS?)

Cinnamic acid esters: $M^{+\bullet}$ 676; key fragments = 133, loss of M-148 (528), 267 or 297, 438, 209, 73
Benzoic acid esters: $M^{+\bullet}$ 650; key fragments = 105, loss of M-122 (528), 267 or 297, 438; 422, 205, 73
Fragments from TMS cinnamates and benzoates: 115, 189, 307 or 337, 268 or 298

NB: the tables above list information compiled from the literature cited (5.5); see 7.5.4 and below for additional data on the compounds present (determined as part of this project).

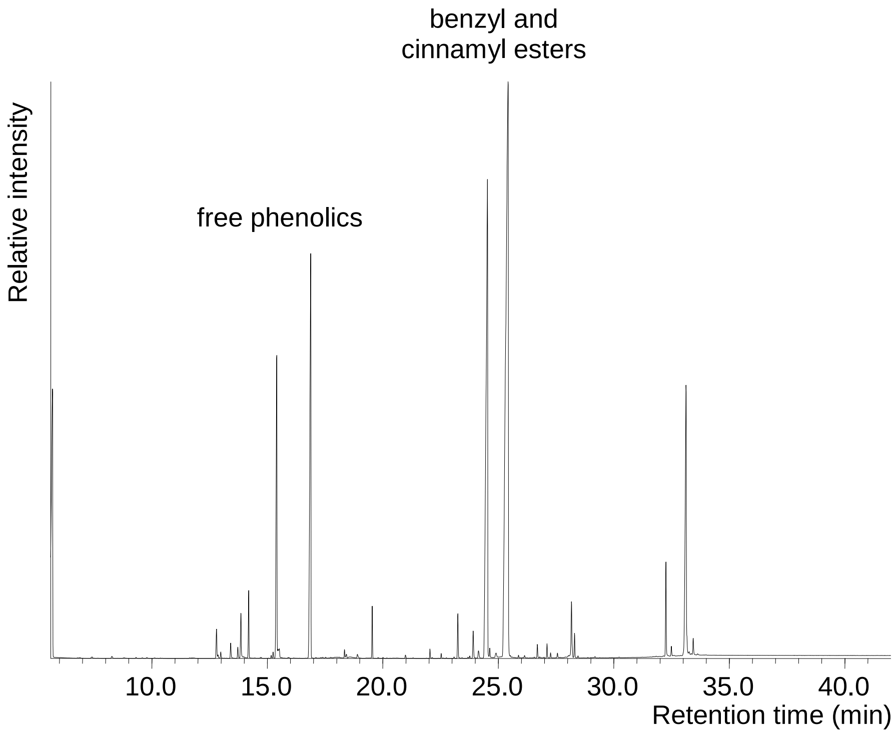


TIC: *Liquidambar orientalis* extract (STOBB)

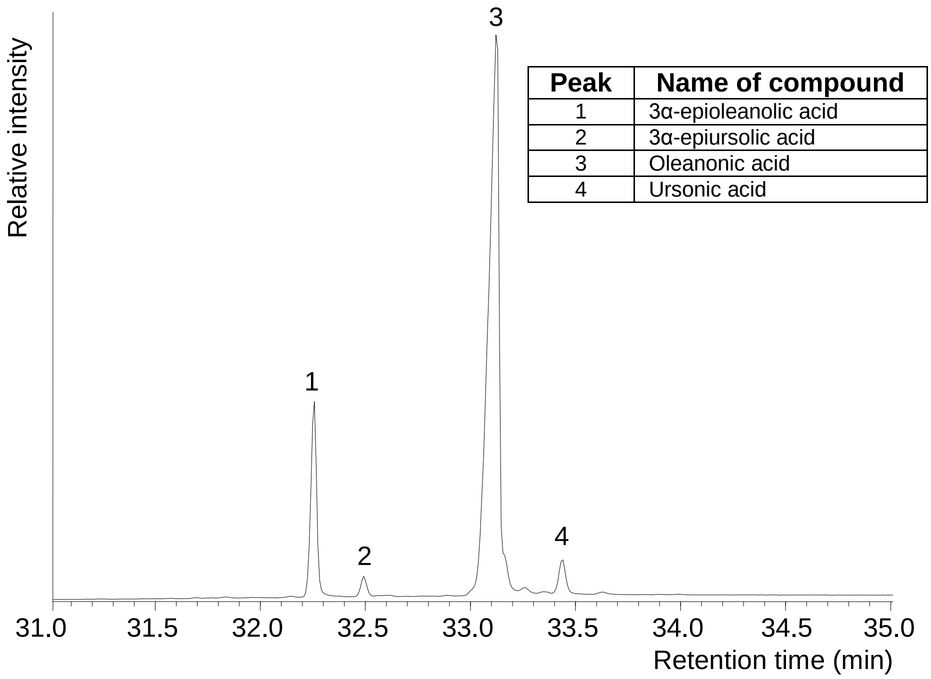


Peak	Name of compound
1	3α-epioleanolic acid
2	3α-epiursolic acid
3	Oleanonic acid
4	3β-oleanolic acid
5	Ursonic acid
6	3β-ursolic acid

Partial TIC: *Liquidambar orientalis* extract (STOBB)

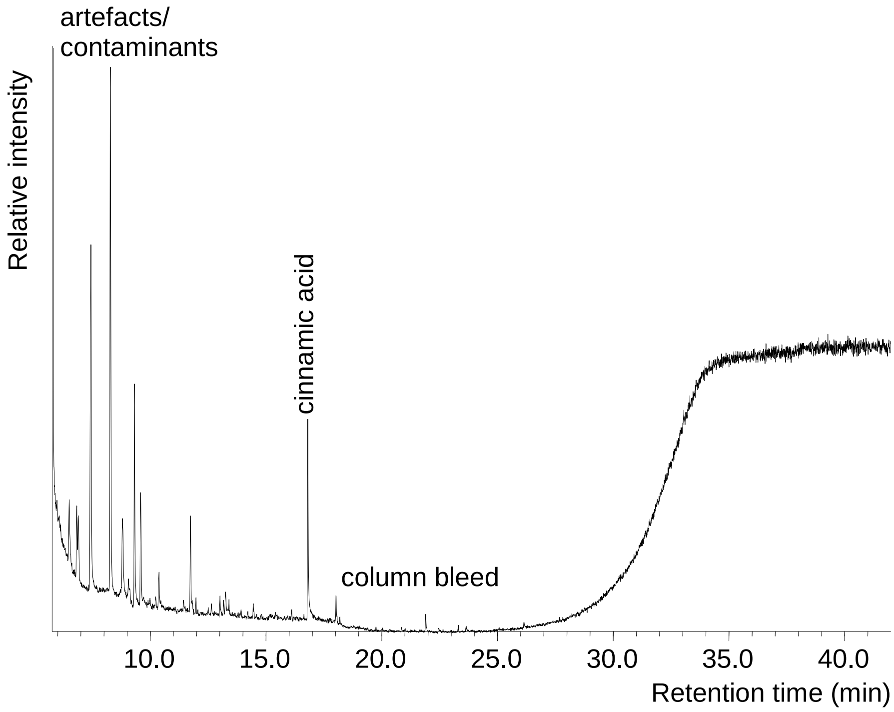


TIC: *Liquidambar orientalis* raw extract (STOSLR)



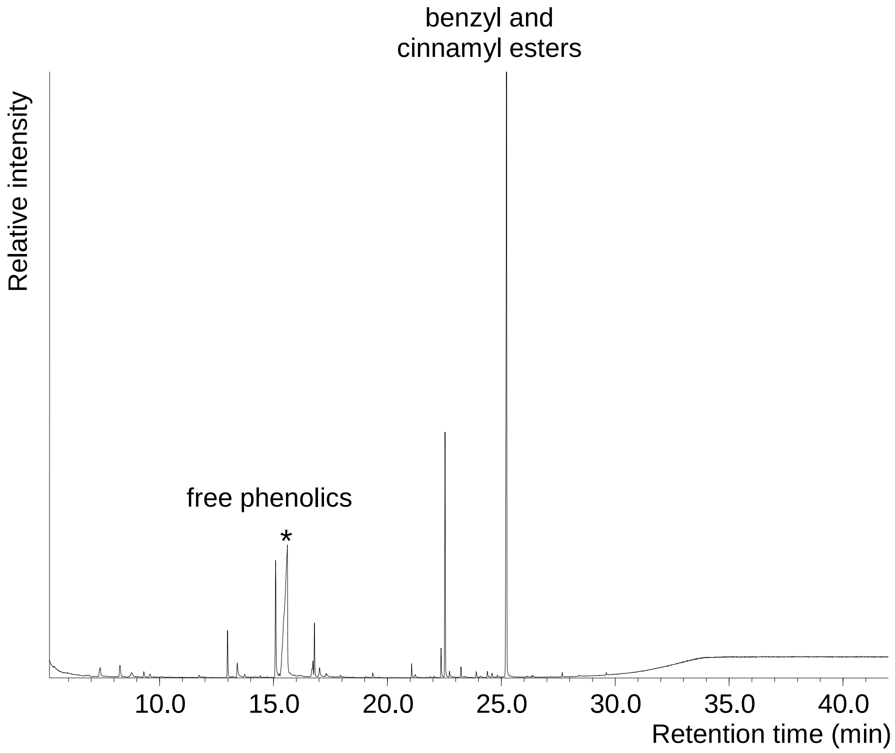
Peak	Name of compound
1	3α-epioleanolic acid
2	3α-epiursolic acid
3	Oleanonic acid
4	Ursonic acid

Partial TIC: *Liquidambar orientalis* raw extract (STOSLR)

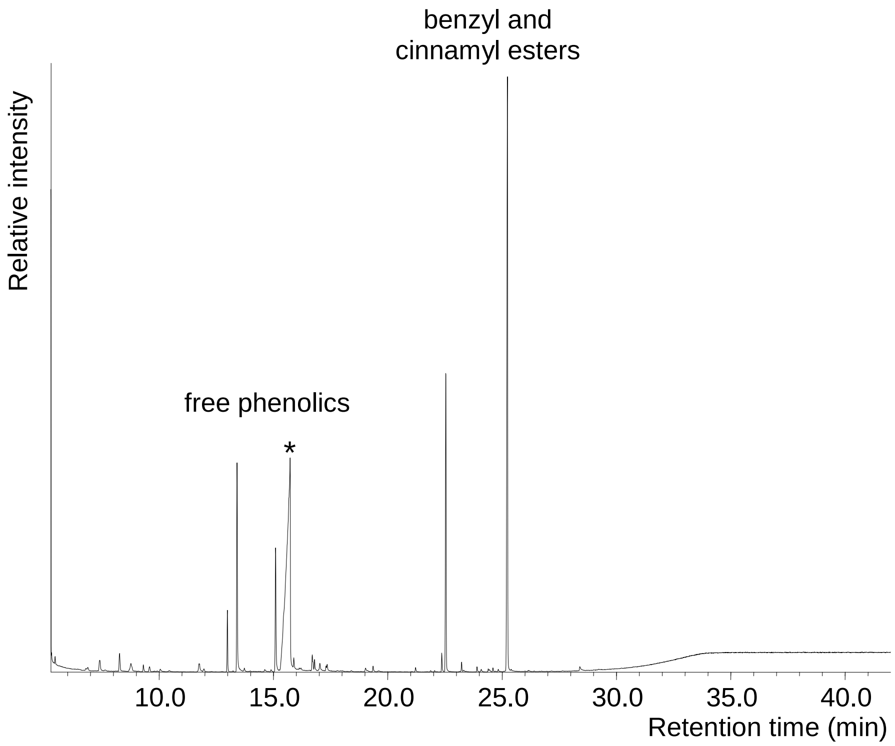


TIC: *Liquidambar orientalis* purified extract (STOSLP)

NB: this sample demonstrates the significant impact of 'purification' on compounds present and indicates that any such pre-treatments have had considerable implications for recovery in the archaeological record

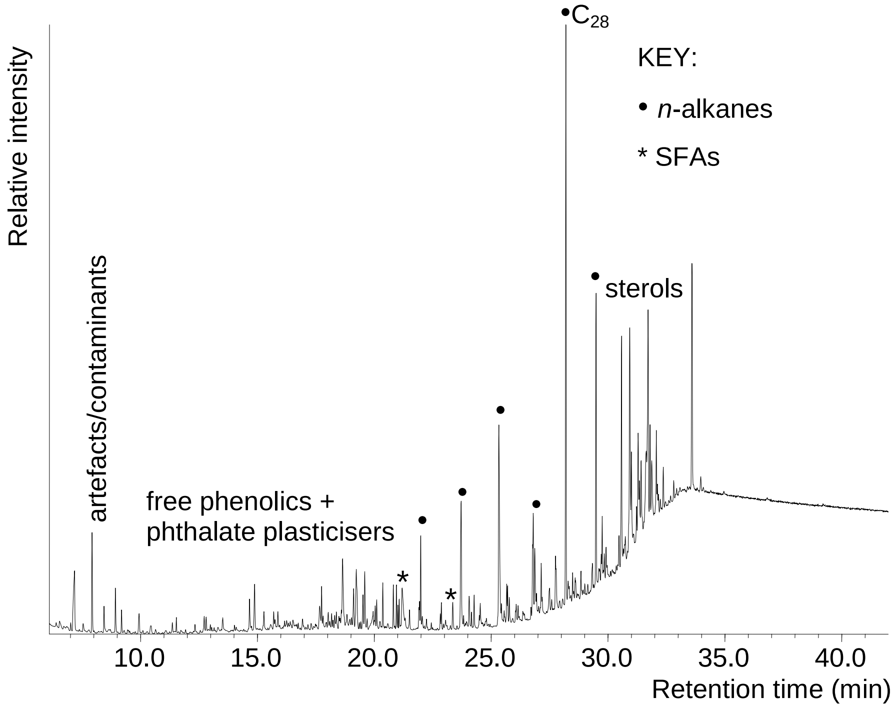


TIC: *Liquidambar orientalis* solidified exudate (STO459)

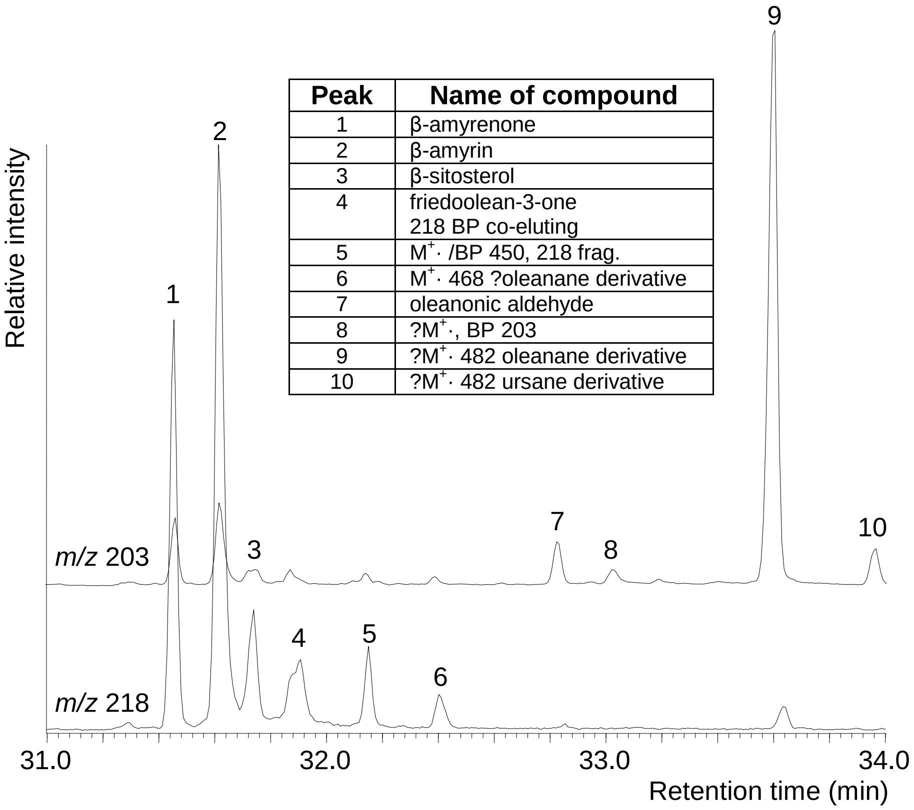


TIC: *Liquidambar orientalis* solidified exudate (STO105)

NB: no traces of triterpenic compounds were observed in these solidified masses which were reported as *Liquidambar orientalis* exudates. May be polymerised (*cinnamic acid only partially derivatised) or the result of natural variability?



TIC: *Styrax officinalis* twig extract (STYBNT)



Peak	Name of compound
1	β-amyrenone
2	β-amyrin
3	β-sitosterol
4	friedoolean-3-one 218 BP co-eluting
5	$M^{+}\cdot$ /BP 450, 218 frag.
6	$M^{+}\cdot$ 468 ?oleanane derivative
7	oleanonic aldehyde
8	?$M^{+}\cdot$, BP 203
9	?$M^{+}\cdot$ 482 oleanane derivative
10	?$M^{+}\cdot$ 482 ursane derivative

XIC: *Styrax officinalis* twig extract (STYBNT)

Appendix 4. Residue analysis of samples from F77, Bezannes.

Report prepared for Denis Bouquin, ReimsMétropole.

Background

The area known as *Le Haut Torchant* on the flanks of Mont Benoît, Marnes, Champagne-Ardenne has revealed evidence of occupation since pre-historic times. By the Iron Age, this region lay in the territory of the Gallic tribe, the *Remi*, who founded the *oppidum* at Vieux-Reims c. 80 BC. As this group chose to ally themselves with Rome during the campaigns of Julius Caesar, this area flourished in the Roman period with a planned town, *Durocortorum Remorum* (Reims) laid out in the reign of Augustus. This foundation became the capital of *Galla Belgica* (King 1990: 72). Recent excavations at Bezannes to the south-west of Reims have uncovered a sequence of ditched enclosures of a mortuary nature. The final phase of use, dated to the 2^{nd}-4^{th} centuries AD, was represented by six single burials and one double burial (F38) of an adult and infant (c. 1 year old).

Most of these individuals had been interred supine, extended in rectangular wooden coffins. Skeletal preservation was poor although indications for the use of shrouds were noted in two graves (F38, F81) while F17 contained hobnails denoting footwear. Other grave goods consisted of a ceramic vessel (F81), rings made of bone (F38) and a copper-alloy ring (F208). In addition, four pits were identified (F14, F30, F31, F126). These probably contained small wooden boxes and, although no human remains were present, two ceramic (F30, F126) and one glass vessel (F31) were recovered (Bontrond and Bouquin 2012). The latter was found to contain an interesting mixture of calcium carbonate, hydrolysed animal fat and a degraded plant oil and/or wax (Garnier 2012).

One individual (F77) had, however, received more elaborate treatment than the norm. This adult of indeterminate sex had been encased in plaster (a generic term for lime plaster or gypsum) and placed in an elaborately decorated lead coffin with tin-soldered corners. Dated to the 3^{rd}-4^{th} century AD, the burial was lifted in its entirety for excavation within a laboratory setting. The lead lid was in good condition despite bending due to pressure from above but the sides of the coffin had bowed inwards resulting in the dissociation of all four corners. Gaps had also formed in the middle of the coffin while the distal panel was absent. Nonetheless, the internal plaster casing which surrounded the human remains had helped to preserve evidence of the treatment of the body with textile impressions denoting a shroud or garment. Hobnails demonstrating the presence of shoes placed on or near the feet were also recovered (Bontrond and Bouquin 2012).

Figure 1. Discoloured plaster and black powdery material from below the skeletal remains.

Below the body the plaster showed evidence of discolouration with pink-orange or brown patches accompanied by a black, powdery material (**Figure 1**). The excavators suggested that this might be the result of chemical alteration due to the absorption of body decomposition fluids or the presence of other organic materials. The inclusion of floral tributes or a plant-based litter was also posited due to the abundance of plant remains within the coffin. Pollen analysis of several samples from this blackened layer provided evidence for cereals, nettles and flowering plants, including some exotic species with a possible east African or south Arabian origin (Corbineau 2012).

Recent research has demonstrated the presence of resinous substances in a number of similar late Roman stone sarcophagus and lead-lined coffin burials from Britain (Brettell *et al.* 2014, 2015), France (Devièse 2008), Italy (Bruni and Guglielmi 2005) and the Rhineland (Reifarth 2013: 91-114). As these finds have included frankincense (olibanum) which grows in these geographical areas (Brettell *et al.* 2015; Devièse 2008: 115-131), it seemed possible that the exotic pollen could have been introduced as particulate adhering to a sticky scented substance. Thus, four samples of materials associated with inhumation F77 were submitted for organic residue analysis at the University of Bradford. These consisted of a portion of the plaster from above the skeleton to provide a control (BZ1), a section of plaster from the base of the lead coffin (BZ2/3) and two samples of mixed materials from the area of the right abdomen/arm (BZ4) and lower legs/ankles (BZ5) (**Figure 1**). A solvent wash of a small portion of the mass of hair found in the neck area was also analysed (BZ6). The samples had been packaged in plastic containers for transportation to the UK. The aim of this investigation was:

- to ascertain if any lipids survived within the samples;
- to identify the lipids present;
- to determine whether those associated with/below the human remains differed from those obtained from the overlying plaster;
- to try to establish the source(s) of these lipids and their archaeological relevance.

Method

Sample preparation

The materials were photographed and details recorded (**Appendix 4.1**). A sub-sample of each was solvent extracted in dichloromethane:methanol (DCM:MeOH, 2:1, *v/v*, 3 x 2 ml) aided by ultrasonication. The solvent-soluble fractions were combined and excess solvent evaporated under a stream of nitrogen. To promote separation, silyl derivatives were produced through trimethylsilylation of each dry residue using ~0.05 ml of *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% TMCS (40 °C, 15 min). Excess reagent was removed by evaporation at room temperature and the derivatised samples re-diluted in DCM (~0.1 ml) for analysis by gas chromatography-mass spectrometry (GC-MS). Disposable screw-topped glass vials were used throughout to minimise cross-contamination. All glass and metal wares were triple-cleaned with dichloromethane prior to use.

GC-MS analysis

The analysis was carried out by combined gas chromatography-mass spectrometry using an Agilent 7890A GC system, fitted with a 30 m x 0.25

mm, 0.25 μm DB-5MS UI 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple axis mass selective detector. The splitless injector and interface were maintained at 300 °C and 280 °C respectively and the carrier gas, helium, at constant flow. The temperature of the oven was programmed to rise from 50 °C (isothermal for 2 min) to 350 °C (isothermal for 10 min) at a gradient of 10 °C per minute. The column was directly inserted into the ion source where electron impact (EI) spectra were obtained at 70 eV with full scan from *m/z* 50 to 800 amu.

Results and discussion

The results are presented as total ion current (TIC) chromatograms of the silylated solvent extracts (**Figures 3-4; Disc 1, File 4.1**). Each separated component is shown as a discrete peak with the area beneath representative of its relative abundance. The components identified have been labelled.

The pure white plaster sample from above the human remains but not in contact with them (BZ1) selected as a control contained no lipid components (the very low abundance peaks are analytical artefacts and a phthalate plasticiser that are only visible due to the absence of other compounds). This suggests that, despite the damage to the lead coffin, the plaster had largely protected the inhumation from the ingress of organic matter from the surrounding soil environment.

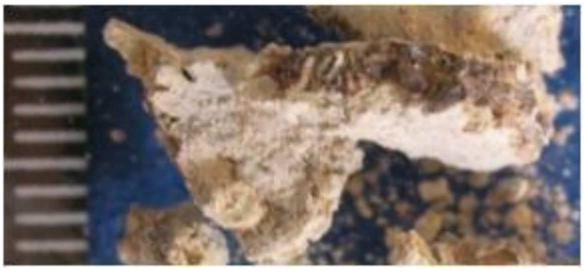


Figure 2. Sub-sample of plaster from below the skeleton (BZ2) showing dark inner layer.

The plaster from the base of the lead coffin and so below the skeleton was sub-sampled (BZ2 and BZ3) as a darker line was noted running through some of the material (**Figure 2**). This 'sandwich' effect has been noted in similar burials from Poundbury Camp, Dorset (Brettell 2012). These sub-samples contained a series of low abundance lipid components (**Figure 3**). Phthalate plasticisers and other modern synthetic contaminants (P) were noted. These probably derive from the plastic packaging in which the samples had been stored. The compounds of interest consisted of saturated straight-chain alcohols (*n*-ols) with an even over odd predominance (EOP), saturated carboxylic acids (SFAs, EOP) with a maximum at $C_{16:0}$ and traces of monounsaturated carboxylic acids (MUFAs, $C_{16:1}$, $C_{18:1}$). These moieties are end-products of the degradation of plant and animal tissues with a contribution from both indicated here due to the presence of cholesterol (ch), diagnostic of mammalian input sources, and β-sitosterol (β), characteristic of plant matter. They cannot, however, be assigned a specific origin and are ubiquitous in nature.

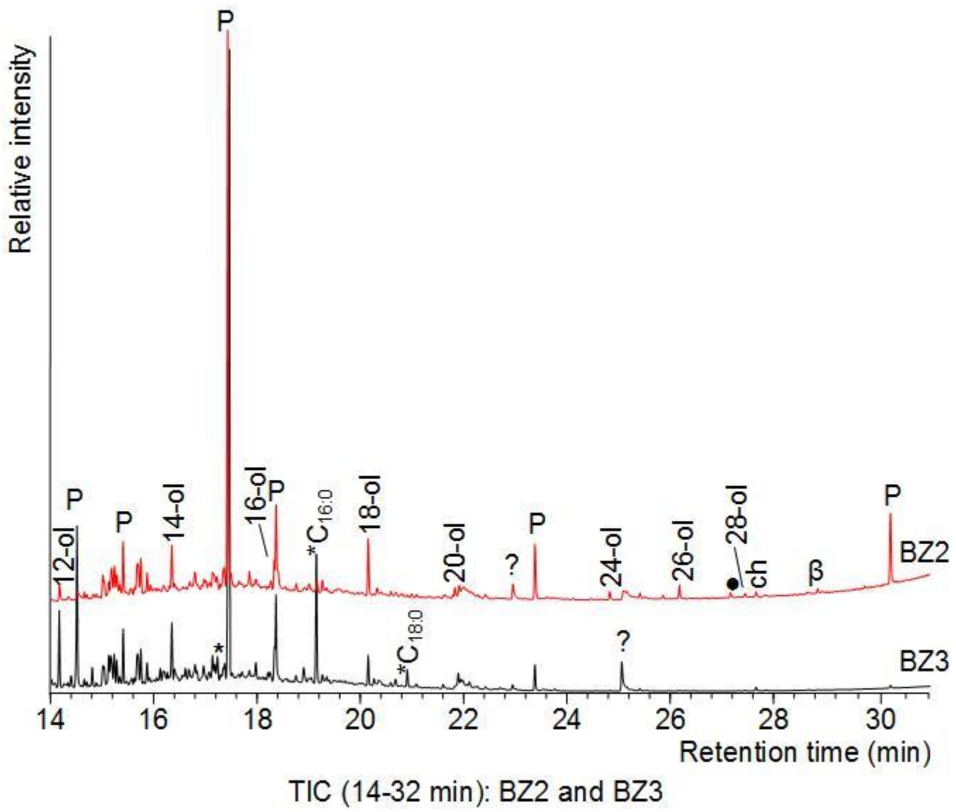


Figure 3. Partial total ion current chromatogram showing key compounds in BZ2 and BZ3. **Key**: plasticisers (P); n-alkanes (●); n-alkanols (XX-ol); SFAs (*$C_{XX:0}$); cholesterol (ch); β-sitosterol (β).

The samples from the right arm/abdomen (BZ4) and lower legs/ankles (BZ5) contained a broader range of lipids (**Figure 4; Table 1**). The extensive series of *n*-alkanols (C_{12-32}) and SFAs ($C_{12:0-28:0}$, $C_{16:0}$ max) in conjunction with diols (C_{20-26}, EOP) and wax esters (C_{40-46}, EOP, C_{40} max) denotes a significant input from epicuticular leaf waxes (Otto *et al*. 2005; Rieley *et al.* 1991). The majority of the steroidal components were also found to be phytosterols (campesterol (cp), brassicasterol (br), stigmasterol (sg), β-sitosterol (β)) and their derivatives confirming the considerable abundance of higher plant matter in these samples. In addition, friedoolean-3-one (7) and ?friedoolean-3-ol (6) were observed.

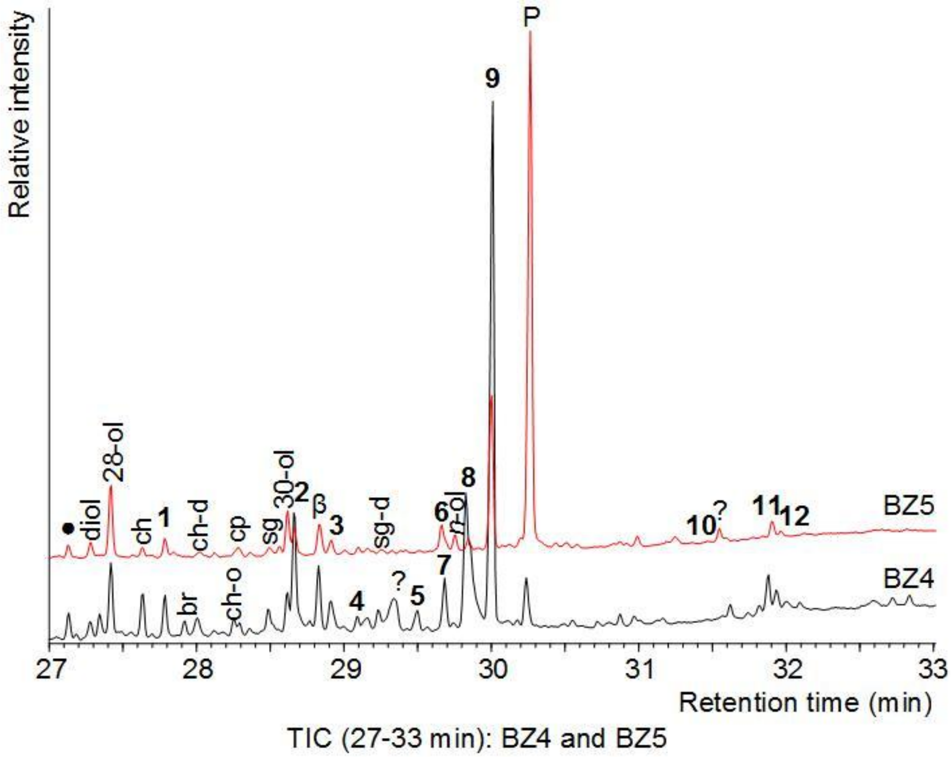


Figure 4. Partial total ion current chromatogram showing key compounds in BZ4 and BZ5. The numbered identifiers refer to **Table 1**. **Key**: *n*-alkanes (●); *n*-alkanols (XX-ol); cholesterol (ch); brassicasterol (br); cholesta-3,5-dien-7-one (ch-d); cholestenone (ch-o); campesterol (cp); stigmasterol (sg); β-sitosterol (β); stigmasta-3,5-dien-7-one (st-d).

Table 1. Molecular ion, base peak and fragment ions, terpenic compounds in BZ4 and BZ5

No.	RT	$M^{+\cdot}$	BP	Key fragment ions	Compound
1	27.8	384	191	369, 281, 207, 177, 109	C_{28} αβ-hopane
2	28.7	410	191	395, 189, 109	diploptene
3	28.9	412	191	398, 281, 177, 109	C_{30} αβ-hopane
4	29.0	498	161	483, 455, 365, 319, 229, 189, 175, 135, 121	?terpenic alcohol
5	29.5	?	218	439, 411, 275, 257, 203>189, 175, 161, 121	oleanane derivative
6	29.6	514	73	499, 409, 273, 205, 189, 159, 143, 121, 109	terpene derivative
7	29.7	468	69	453, 425, 273, 218, 205, 189, 175, 161, 109	terpene derivative
8	29.8	500	95	485, 347, 237, 205, 191, 177, 109	?friedoolean-3-ol
9	30.0	426	69	411, 273, 218, 205, 191, 175	friedoolean-3-one
10	31.5	?454	233	439, 369, 215, 191, 109	
11	31.8	?454	191	439, 367, 233, 217, 109	
12	31.9	?	191	453, 369, 247, 229, 205, 177	

These pentacyclic triterpenic compounds occur in the tissues of a number of plant families, principally members of the Celastraceae, Hippocrateaceae and Euphorbiaceae (Shan *et al*. 2013). The MUFAs and LMM SFAs ($C_{12:0-}$

$_{18:0}$), could derive from the same source and/or from degraded animal tissues. A contribution from the latter is supported by the presence of cholesterol and its derivatives while diploptene (2) is considered to be a marker for the bacterial degradation of bone and has previously been observed in similar burial contexts (Brettell 2013; Evershed *et al.* 1995; Green 2013). Traces of hopanes may also reflect bacterial input (Greenwood *et al.* 2006). The most parsimonious explanation is that these low abundance compounds derive from the decomposition of the human remains. In contrast, the dominant contribution from degraded plant matter indicates the deposition of floral tributes and, perhaps, other materials. This corresponds closely with the findings of Corbineau (2012) who identified pollen from a wide variety of taxa in samples associated with the skeletal remains.

Figure 5. Glass vessel recovered from pit F31, Bezannes. Image ©Garnier 2012, Figure 1.

Unfortunately, no indications for the presence of resinous substances were recovered and, in the absence of specific biomarkers, further interpretation is unwise. It should be noted, however, that the use of essential oils, gums or a gum-resin such as myrrh cannot be ruled out as such exudates only survive over archaeological time in exceptional circumstances. The similarities between the results discussed here and those obtained from analysis of the residue in the 'biberon' from pit F31 (**Figure 5**) are also intriguing. This correspondence may be an artefact of the homogenising effect of the degradation pathways of plant and animal tissues which result in a limited number of end products. Thus, the surviving lipid content in both of these contexts (and many others) tends to be alike although at Bezannes they do appear to derive from anthropogenic actions rather than soil ingress.

The presence of calcium carbonate within the glass vessel (unless this is ingress from the burial environment) could also connect this find not only with the rites used in the treatment of the individual in F77 but with wider research into substances deemed appropriate for use in Roman period mortuary rites (Brettell *et al.* 2014; Schotsmans 2013: 7-13, 191-204). In a number of instances, it is now clear that resinous substances were incorporated within the textiles wrapped around individuals interred in lead-lined coffins and stone sarcophagi, a number of whom had been encased in plaster (Brettell *et al.* 2015; Reifarth 2013: passim). There is clear no evidence for this form of 'embalming' here but there is the possibility that scented oils, applied to the skin/textile wrappings or poured over the body as libations, may have played a role and could have been transported in glass vessels. If the aromatic components of such a mixture comprised low molecular mass volatiles they would have been rapidly lost leaving only traces of the pressed plant oil/leaf wax matrix. The deliberate burial of this glass 'biberon' and two ceramic vessels in wooden boxes within this mortuary context could provide support for this view and represent the ritual deposition of these containers. Much more work is required, however, to substantiate such a claim.

The solvent extract of a small portion of the hair contained a range of SFAs and *n*-alkanols with traces of LMM aromatics and nitrogen-containing compounds. Low levels of cholesterol, diploptene and a number of phytosterols (campesterol, stigmasterol, β-sitosterol) were also observed. Extensive research has shown that the surface and internal components of human hair consist of c. 50% carboxylic acids (C_{12-20}), principally $C_{16:0}$ (18%), $C_{18:0}$, $C_{16:1}$ and $C_{18:1}$ together with wax esters, hydrocarbons and traces of squalene, cholesterol, triacylglycerides and ceramides (waxy lipids composed of di- and tri-hydroxy long-chain bases linked via amide bonds to saturated carboxylic acids up to C_{24} in length). The remaining c. 40% consists of 18-methyl eicosanoic acid (18-MEA) which is bonded via thioester linkages to epicuticular proteins (Robbins 2012: 130-145). Thus, the compounds present are clearly consistent with moieties derived from the sebaceous glands and matrix cells of human hair with the addition of some adhering plant matter (phytosterols) and diploptene denoting the bacterial degradation of bone.

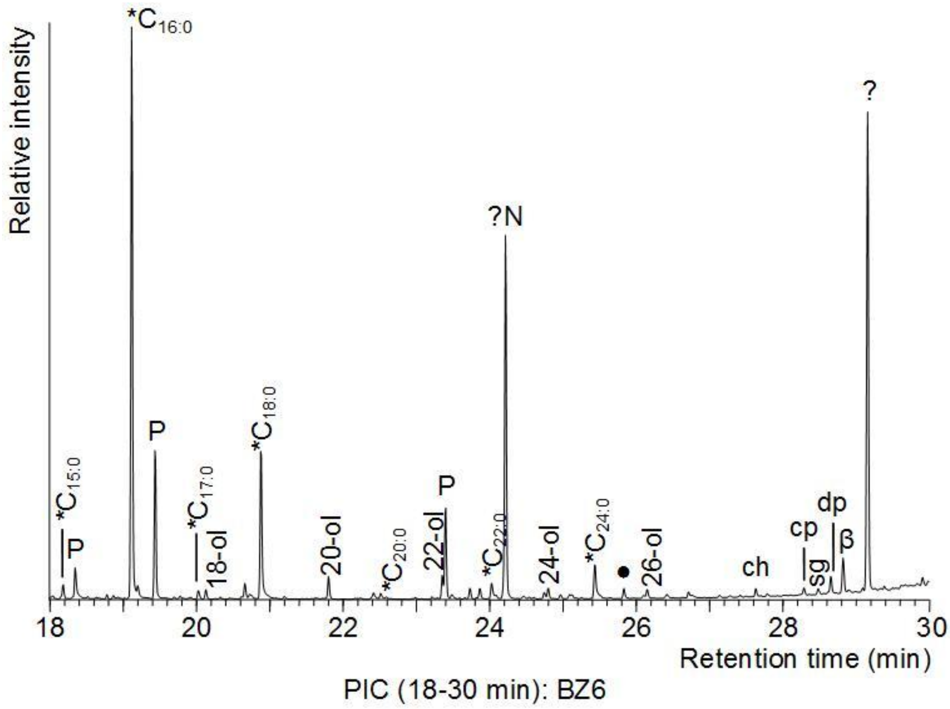


Figure 5. Partial TIC showing compounds in BZ6. **Key**: *n*-alkanes (●); *n*-alkanols (XX-ol); SFAs (*$C_{XX:0}$); cholesterol (ch); campesterol (cp); stigmasterol (sg); diploptene (dp); β-sitosterol (β); nitrogenous compound (?N); unidentified compound (?; **Appendix 4.2**).

Conclusion

Molecular analysis of samples obtained from a late Roman period inhumation interred in a decorated lead coffin at Bezannes, near Reims, France have shown the presence of degraded plant and animal matter. The compounds identified cannot be associated with any specific source and most are often found in soils. In this instance, however, the secure context and absence or low abundance of moieties in the plaster samples from above and below the body suggests that the lipids in the dark matter associated with the skeletal remains are of archaeological relevance. These largely derive from the breakdown of the tissues of higher plants which, in conjunction with the pollen data, suggests that deliberate deposits in the form of floral tributes and/or a plant-based litter were made at the time of burial. No evidence for the inclusion of resinous exudates were observed so no clear explanation for how exotic pollen became incorporated within this burial can be offered.

Nonetheless, the use of a plant gum or gum-resin such as myrrh to anoint the body or the application of a libation consisting of a plant oil scented with volatile aromatic compounds cannot be ruled out as traces of these would only survive in exceptional circumstances in the archaeological record.

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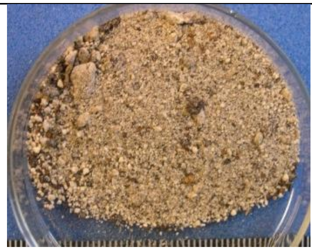
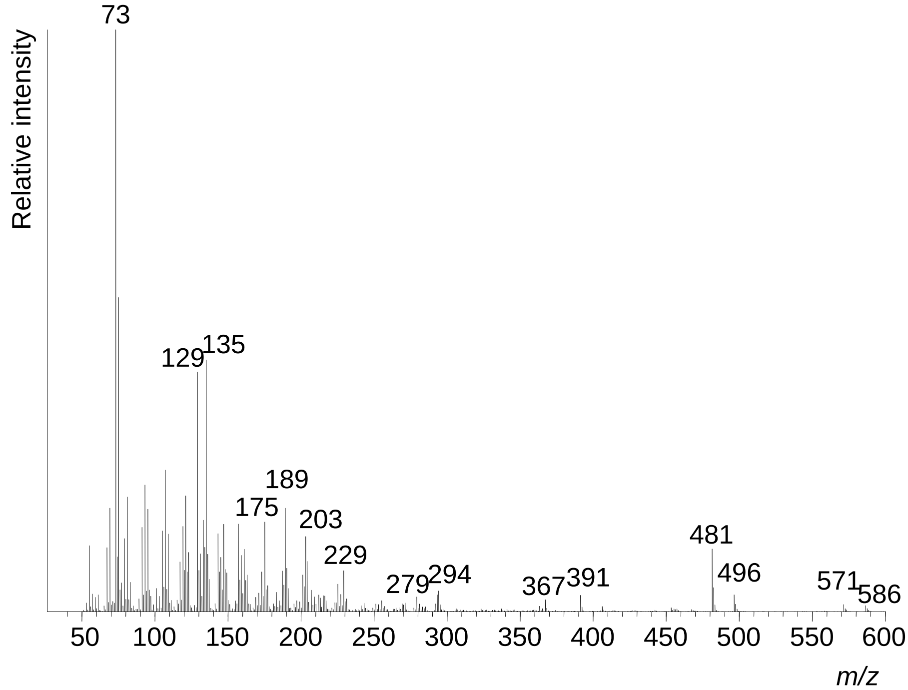
Bezannes, Appendix 4.1. Table providing details of the samples from F77, Bezannes

ID	Sample Code	Mass (g) SE	Image	Description	Compounds present
BZ1	6903 F10.09 en avant du squelette	4.0		Pure white plaster A portion of the plaster covering the skeleton but not in contact with it	**No organic matter**
BZ2	6903 F10.09 sous le sarcophagus	4.0		Plaster with dark inner layer A portion of the plaster beneath the skeleton from the base of the lead coffin	**Degraded plant/animal matter** ***n*-alkanols**: $C_{12\text{-}28}$ (EOP, bimodal, C_{18} + C_{26} max) **SFAs**: $C_{14:0\text{-}18:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; β-sitosterol
BZ3	6903 F10.09 sous le sarcophagus	4.0		Plaster with darker inclusions A portion of the plaster beneath the skeleton from the base of the lead coffin	**Degraded plant/animal matter** ***n*-alkanols**: $C_{12\text{-}18}$ (EOP, C_{12} max) **SFAs**: $C_{12:0\text{-}18:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; β-sitosterol
BZ4	6903 F10.09 C. D1	4.0		Mixture of plaster and dark materials Material from the area of the right forearm/right lateral section of the abdomen	**Degraded plant and animal matter** ***n*-alkanes:** traces (C_{28} max); ***n*-alkenes**: $C_{21\text{-}24}$-dienes ***n*-alkanols:** $C_{12\text{-}30}$ (EOP) **SFAs:** $C_{12:0\text{-}28:0}$ (EOP, $C_{16:0}$ max); **branched** $C_{15:0;\ 17:0}$ **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols/stanols**: cholesterol; coprostanol; brassicasterol; cholest-3,5,-dien-7-one; cholestenone; campesterol; stigmasterol; β-sitosterol; stigmasta-3,5,-dien-7-one **Terpenes:** diploptene; ?friedoolean-3-ol; friedoolean-3-one + related compounds **Wax esters**: $C_{40\text{-}46}$ (EOP, C_{40} max)

BZ5	6903 F10.09 C. J2	4.0		Mixture of plaster and dark materials Material from the area of the lower legs/ankles	**Degraded plant and animal matter** ***n*-alkanes:** traces ***n*-alkanols:** C_{12-32} (EOP); **diols**: C_{20-26} (EOP) **SFAs:** $C_{14:0-24:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols/stanols**: cholesterol; campesterol; stigmasterol; β-sitosterol; stigmastanol; stigmasta-3,5,-dien-7-one **Terpenes:** diploptene;?friedoolean-3-ol; friedoolean-3-one + related compounds **Wax esters**: C_{40-46} (EOP, C_{40} max)
BZ6	6903 F10.09 Hair	NA		Solvent wash of portion of dark brown human hair Portion of hair from neck area of the skeleton	**Surface/internal hair components + plant matter** Benzene/phenol derivatives; nitrogenous compounds ***n*-alkanols:** C_{16-26} (EOP, C_{20} max) **SFAs:** $C_{12:0-26:0}$ (EOP, $C_{16:0}$ max); **branched** $C_{15:0-17:0}$ **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols/stanols**: cholesterol; campesterol; stigmasterol; β-sitosterol; stigmastanol **Terpenes:** diploptene; unidentified compound

Total ion current (TIC) chromatograms of the silylated samples can be found on **Disc 1, File 4.1**.

Bezannes, Appendix 4.2. Mass spectrum of unidentified compound in hair sample



Mass spectrum (29.2 min): BZ6

The fragmentation pattern is indicative of a polyaromatic hydrocarbon with hydroxy and/or carboxylic acid functional groups (73 base peak denoting loss of TMS group(s)). It is almost certainly a terpenoid or steroid derivative.

Appendix 5. Residue analysis of Egyptian mummies (Brettell *et al.* 2015c)

5.1 Pilot study: analysis of materials detached from Ancient Egyptian mummies.

Prepared for Dr L. McKnight and Dr S. Atherton, Ancient Egyptian Bio Bank Project, University of Manchester, UK

Background

Materials detached as a result of wear and tear from Egyptian mummies have often been discarded or collected and stored without further investigation. As these artefacts are so precious, samples are rarely available for chemical analysis even using minimally destructive techniques such a gas chromatography-mass spectrometry. This debris may, therefore, have considerable value in illuminating questions regarding embalming techniques. In order to assess the potential of this approach, materials which had fallen from the wrappings of thirteen votive animal mummies and one human mummy were submitted for lipid analysis at the University of Bradford (**Table 1**). These materials had been stored in aluminium foil or glass vials at the University of Manchester, although their previous history is not fully known. The aim of this pilot investigation was:

- to ascertain if any lipids survive within samples of this nature;
- to identify the lipids present;
- to establish the probable source(s) of these lipids;
- to determine the minimum sample mass required for meaningful results to be obtained.

Method

Sample preparation

The materials were photographed and details recorded (**Appendix 5.1.1**). A sub-sample of each was solvent extracted in dichloromethane:methanol (DCM:MeOH, 2:1, *v/v*, 3 x 2 ml) aided by ultrasonication. The solvent-soluble fractions were combined and excess solvent evaporated under a stream of nitrogen. To promote separation, silyl derivatives were produced through trimethylsilylation of each dry residue using ~0.5 mL of *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% TMCS (40 °C, 15 min).

Excess reagent was removed by evaporation at room temperature and the derivatised samples re-diluted in DCM (~0.1 ml) for analysis by gas chromatography-mass spectrometry (GC-MS). Disposable screw-topped glass vials were used throughout to minimise cross-contamination. All glass and metal wares triple-cleaned with dichloromethane prior to use.

GC-MS analysis

The analysis was carried out by combined gas chromatography-mass spectrometry using an Agilent 7890A GC system, fitted with a 30 m x 0.25 mm, 0.25 μm DB-5MS UI 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple axis mass selective detector. The splitless injector and interface were maintained at 300 °C and 280 °C respectively and the carrier gas, helium, at constant flow. The temperature of the oven was programmed to rise from 50 °C (isothermal for 2 min) to 350 °C (isothermal for 10 min) at a gradient of 10 °C per minute. The column was directly inserted into the ion source where electron impact (EI) spectra were obtained at 70 eV with full scan from *m/z* 50 to 800 amu.

Results and discussion

The results are presented as total ion current (TIC) and extracted ion current (XIC) chromatograms of the silylated solvent extracts with mass spectra of key compounds (**Figures 1-16; Disc 1, File 5.1**). Each separated component is shown as a discrete peak with the area beneath representative of its relative abundance. The components discussed in the text have been labelled. Assignments have been made through mass spectral interpretations based on the molecular mass and established fragmentation patterns of simple and complex lipids and their relative retention times.

Contaminants

The majority of the samples contained low levels of phthalate plasticisers. These are modern contaminants, probably derived from plastic packaging, which had come in contact with the bundles at some point in their history.

Table 1. Details of the provenance and nature of the Ancient Egyptian mummy samples analysed

GC-MS no.	AEABB no.	Species	Mass (g)	Description	Institution details	Institution reference
MM1	595/1	Cat	0.05	Debris from neck region - sweepings loose in tissue paper	Old Speech Room Gallery, Harrow	
MM2	191/1	Cat	0.06	Linen & debris from head - sweepings	Bristol Museum and Art Gallery	H3065
MM3	464/2	Bird	0.07	Debris from head end - loose in wrappings	Buckinghamshire Museum	AYBCM:2003-91.2
MM4	494/2	Ibis	0.01	Debris - loose in tissue paper	Plymouth City Museum	1920.88
MM5	465/4	Bird	0.03	Linen & debris - loose under abdomen	Buckinghamshire Museum	AYBCM:2003-91.1
MM6	471/5	Ibis	0.02	Debris from base - loose after photography	Elgin Museum	
MM7	144/3	Cat	0.08	Debris - loose in box	Nottingham Museum	NCM 1933-281/1
MM8	001/1	Jackal	0.04	Linen & debris - sweepings loose in tissue paper	Grantham Museum	LCNGR1995.E.657
MM9	150/5	Cat	0.01	Translucent fragments x2 - removed from bag of debris	Derby Museum	DBYMU1929-189/1
MM10	150/5	Cat	0.02	Linen fragments - removed from bag of debris	Derby Museum	DBYMU1929-189/1
MM11	146/2	Hawk	0.005	?Black substance from rear aspect - area of damage	Nottingham Museum	HH-X 1095
MM12	165/2	Ibis	0.1	Debris - removed from packaging	Oriental Museum, Durham University	DUROM.1971.122 Saqqara tomb 3508
MM13	162/2	Ibis	0.05	Dark-coloured resin fragment from foot area - loose	Oriental Museum, Durham University	EG727
MM14	162/2	Ibis	0.05	Piece of ?charred material from foot area - loose	Oriental Museum, Durham University	EG727
MM15	162/1	Ibis	0.05	Amber-coloured resin fragments from foot area - loose	Oriental Museum, Durham University	EG727
MM16	401/4	Animal	0.25	Dark material with feather and textile impressions	Museum of Fine Arts, Boston	
MM17	PM1	Human	0.1	Debris - loose in trough of coffin	Perth Museum and Art Gallery, Scotland	

Levels of significance

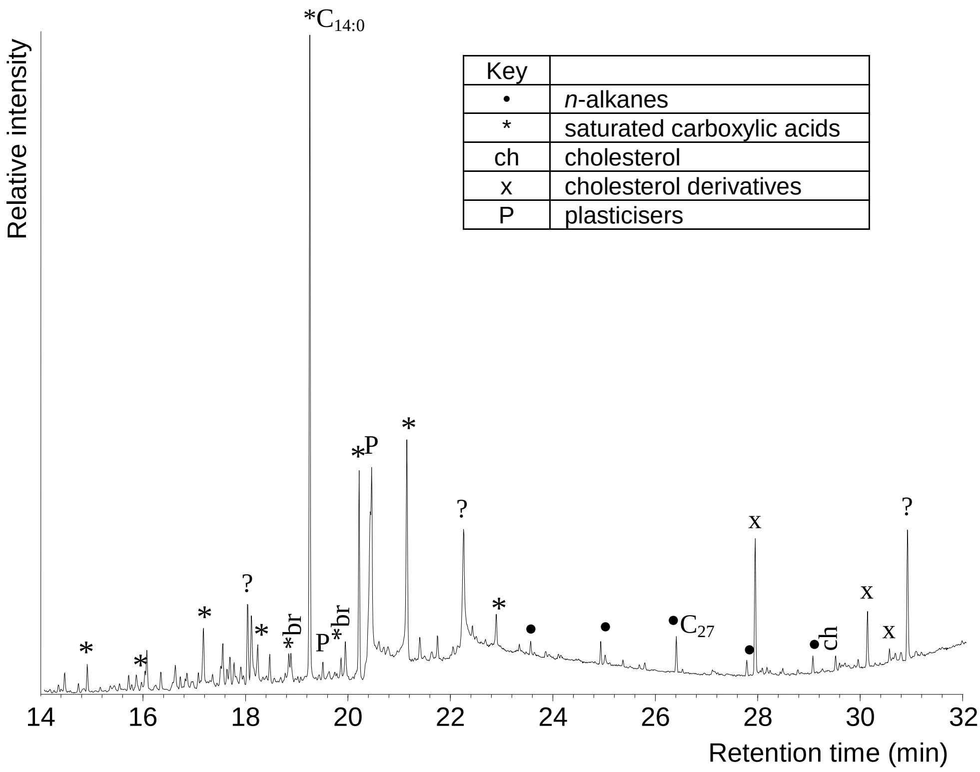
The samples were carefully selected to provide a range of masses in order to assess the minimum amount required for meaningful results to be obtained. A number (n = 5) contained only tiny traces of ubiquitous lipids below the level of instrumental artefacts and so cannot be considered of significance (MM3; MM4; MM5; MM8; MM10). Thus, this approach indicated that where a pure, lipid-rich substance was present >0.05 g provided sufficient material for identification of source and, if appropriate, multiple investigations to be undertaken. However, when the sample consisted of mixed detritus with a considerable contribution from inorganic components and/or degraded textiles an order of magnitude greater (~0.5 g) is desirable although some indication as to the substance(s) present can be obtained from >0.1 g.

Inorganic and charred materials

Examination of the two translucent fragments (MM9) associated with the Derby Museum cat (AEABB 150/5) indicated that they were inorganic and may be some form of silicate. Likewise MM14, the dark material of charred appearance from the Durham University ibis (162/2), was devoid of any extractable organic matter. Similar carbonised plant materials have been reported in human mummies (Maurer *et al.* 2002). These two samples would repay investigation by other instrumental techniques to illuminate their structure and/or chemical composition.

Oils, fats and waxes

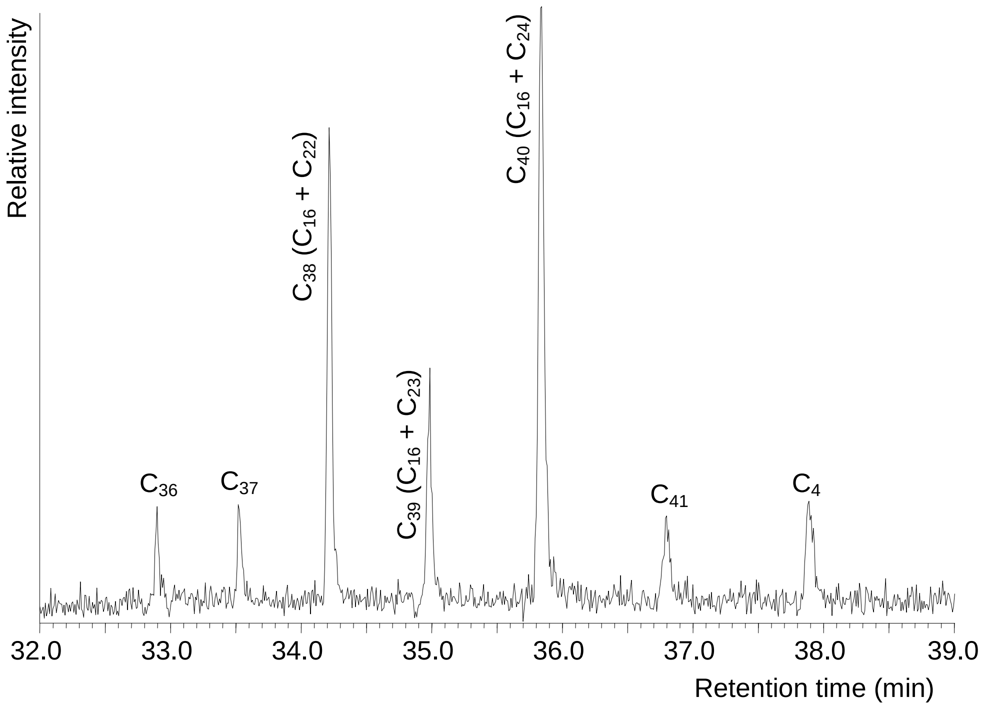
A range of lipid species were observed in MM1 (Harrow, 595/1), MM2 (Bristol, 191/1) and MM7 (Nottingham, 144/3). These three sample extracts were obtained from the degraded textile wrappings of mummified cats. The nature and distribution of the moieties present suggests contributions from a variety of sources. The bimodal distribution of the saturated carboxylic acids (SFAs) may represent a microbial input ($C_{8:0\text{-}11:0}$, $C_{9:0}$ max.) with $C_{12:0\text{-}18:0}$ indicative of a plant oil or animal fat (Evershed 2008). The presence of cholesterol and its derivatives in MM2 and MM7 together with branched-chain carboxylic acids provide some support for the latter and could, therefore, derive from the decomposition of the body or from an applied mammalian fat (Buckley and Evershed 2001; Forbes *et al.* 2002). The high relative abundance of $C_{14:0}$ in these samples is highly unusual and has yet to be explained (**Figure 1**).



TIC (14-32 min): sample MM7, mummified cat, Nottingham, 144/3

Figure 1. Partial TIC of loose material detached from mummified cat, AEABB 144/3

In contrast, the high molecular mass (HMM) *n*-alkanes with an odd-over-even (OEP) carbon number predominance (C_{23}-$C_{33,}$ C_{27} max.) and the long-chain monoesters of palmitic acid ($C_{36\text{-}42,}$ C_{40} max.) observed in MM2 (**Figure 2**) could derive from the epicuticular waxes of higher plants (Rieley *et al.* 1991). Beeswax is an alternative source particularly as the *n*-alkane maximum falls at C_{27}. This substance has previously been identified in both human and animal mummies (Buckley and Evershed 2001; Buckley *et al.* 2004) but is generally characterised by wax monoesters greater than 40 carbons in length (C_{46} max.) together with their degradation products, HMM *n*-alkanols and carboxylic acids (Garnier *et al.* 2002; Heron *et al.* 1994). These compounds were not observed although it should be noted that an absence of *n*-alkanols and reduction in ester components has been reported in experimentally heated beeswax (Namdar *et al.* 2009).

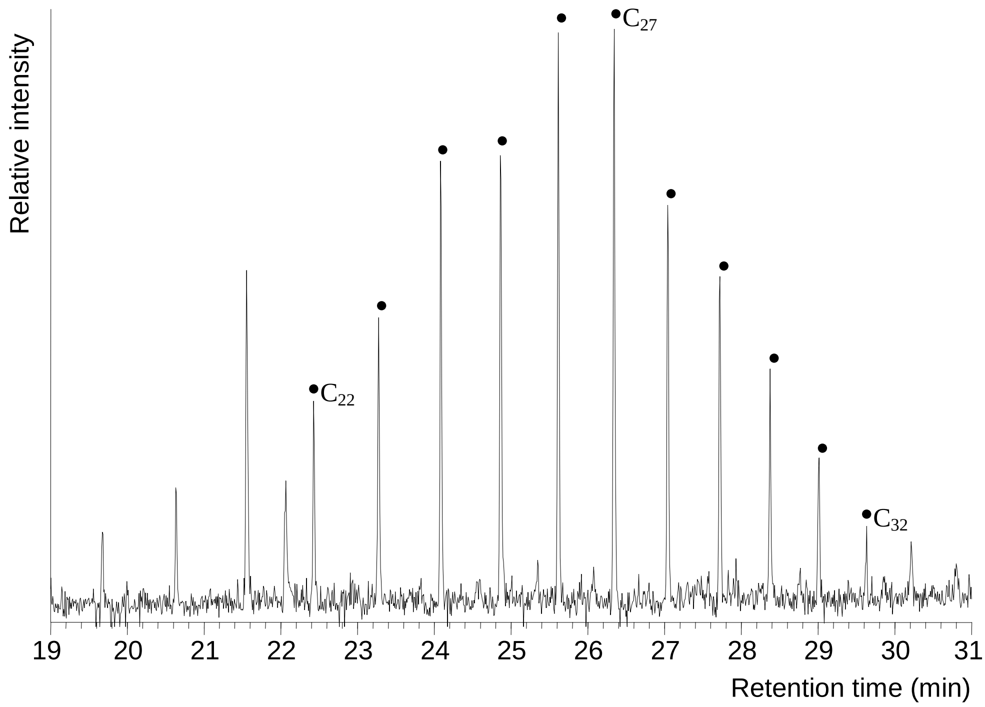


XIC (*m/z* 257): sample MM2, mummified cat, Bristol, 191/1

Figure 2. Partial XIC, wax esters in material from cranium of mummified cat, AEABB 191/1

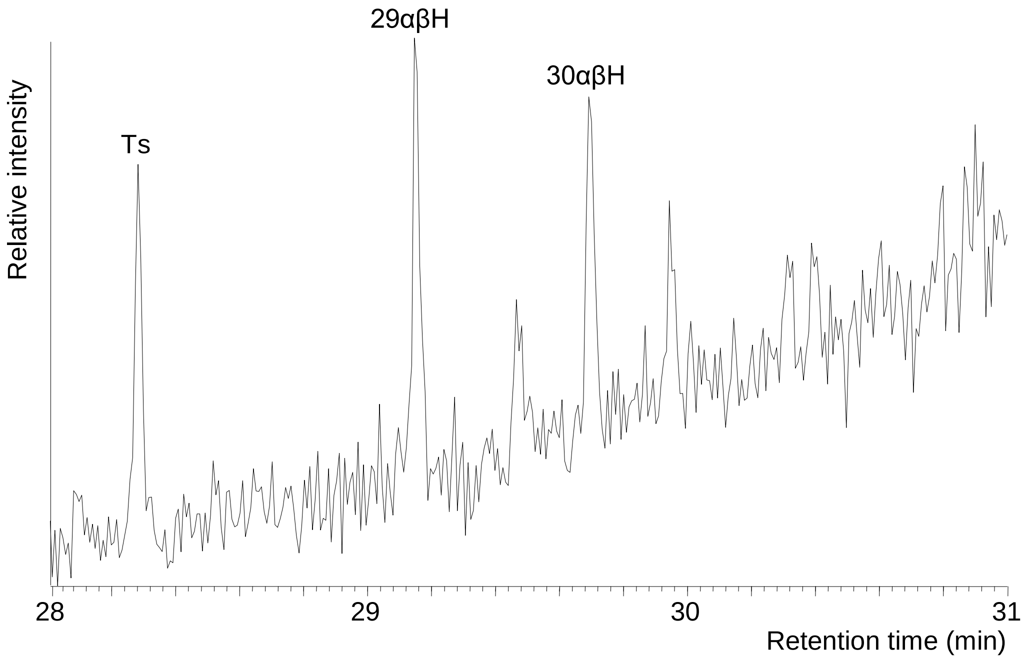
Fossil hydrocarbons

The glossy black 'droplets' associated with the mummified hawk from Nottingham (146/2) contained an homologous series of *n*-alkanes (**Figure 3**) alongside traces of hopanes (**Figure 4**). Key compounds such as pristane and phytane were not observed but the *n*-alkane pattern suggested that this substance could derive from a waxy oil in the early stages of maturity, possibly a type III kerogen or coal (Hunt 1996: 402; Killops and Killops 2005: 381). The latter is more likely as the major components in the TIC are aromatic intermediate and naphthenic hydrocarbons which are commonly found in terrestrial coal-sourced crude oils (Hunt 1996: 329-331, 404-405; Killops and Killops 2005: 150-153; **Figure 5**). Indeed, assessment of certain diagnostic ratios (AN/PH+AN = 0.09, <0.1 petroleum; FLT/FLT+PYR = 0.54, >0.5 pyrogenic; BaA/BaA+CHR = 0.5, asphalt/crude oil combustion) is consistent with the combustion of a bituminous coal (Brändli *et al.* 2006*;* Dong *et al.* 2012; Yunker *et al.* 2002). The absence of expected biomarkers such as steranes and T_m (17α(H)-22,29,30-trisnorhopane) may also be due to the material having been heated prior to its application as T_m is less thermally stable than T_s (18α(H)-22,29,30-trisnorneohopane).



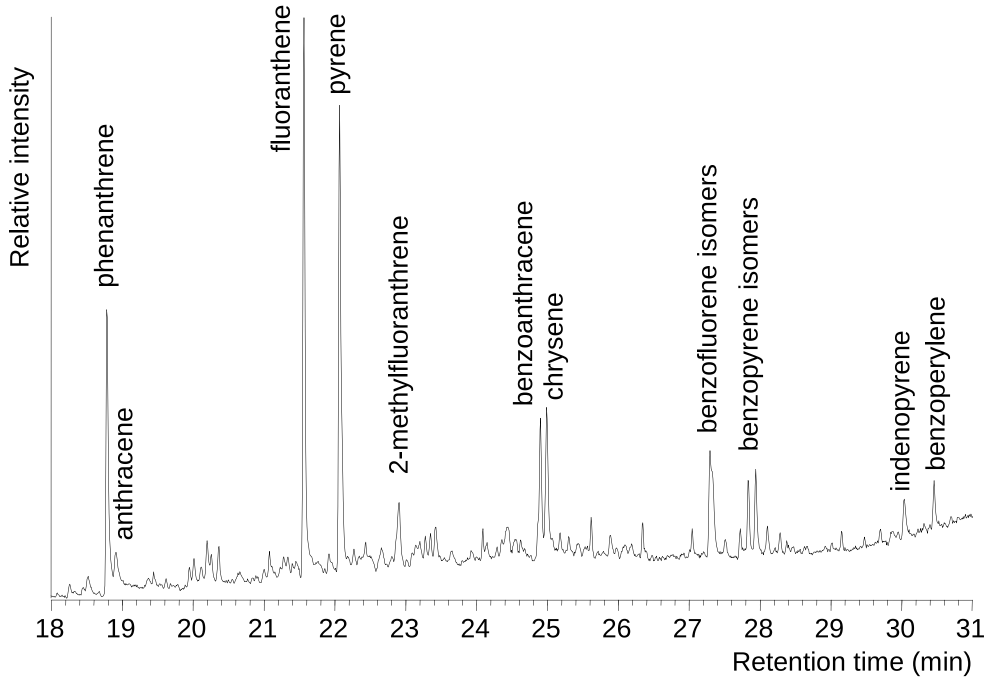
XIC (*m/z* 85): sample MM11, mummified hawk, Nottingham, 146/2

Figure 3. Partial XIC, *n*-alkanes in black substance, mummified hawk, AEABB 146/2



XIC (*m/z* 191): sample MM11, mummified hawk, Nottingham, 146/2

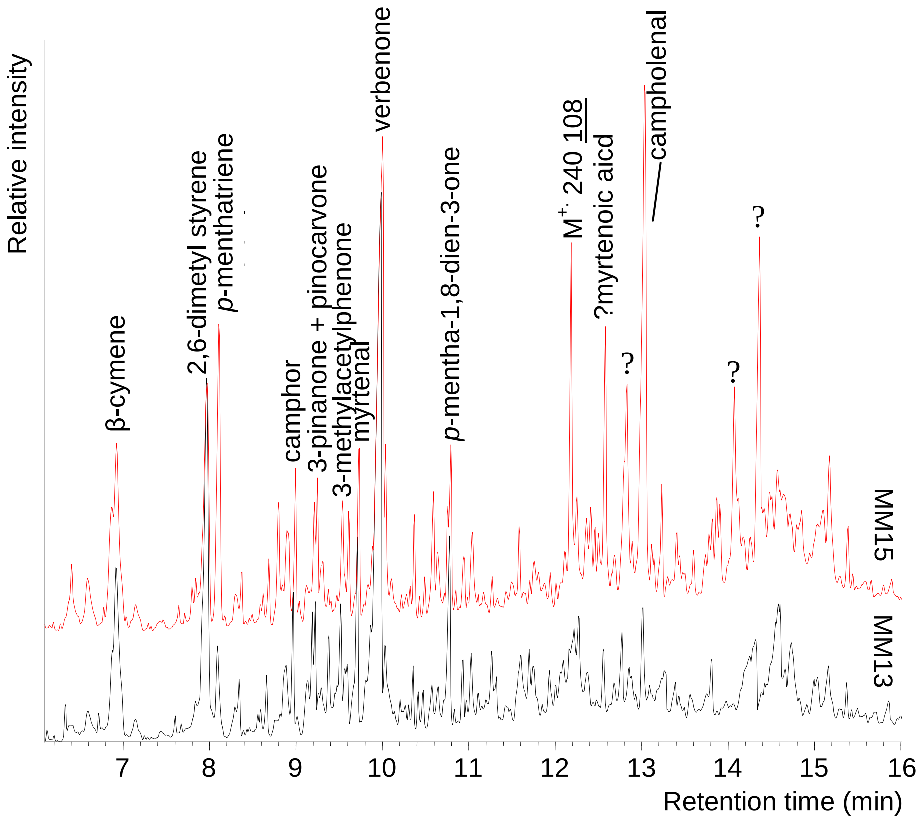
Figure 4. Partial XIC, hopanes in black substance, mummified hawk, AEABB 146/2



TIC (18-31 min): sample MM11, mummified hawk, Nottingham, 146/2

Figure 5. Partial TIC, polyaromatic hydrocarbons, black substance, mummified hawk, AEABB146/2

Plant resin

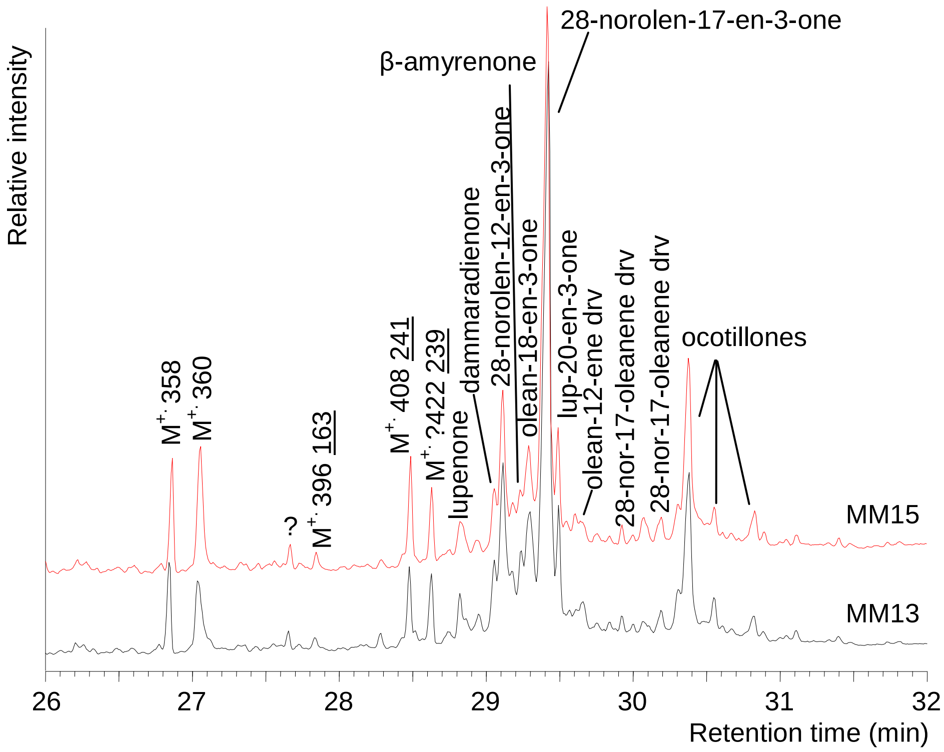


TIC (6-16 min): mummified ibis, MM13 & MM15, Oriental Museum, Durham, 162

Figure 6. Partial TIC, sesquiterpenes in samples from mummified hawk, AEABB 162

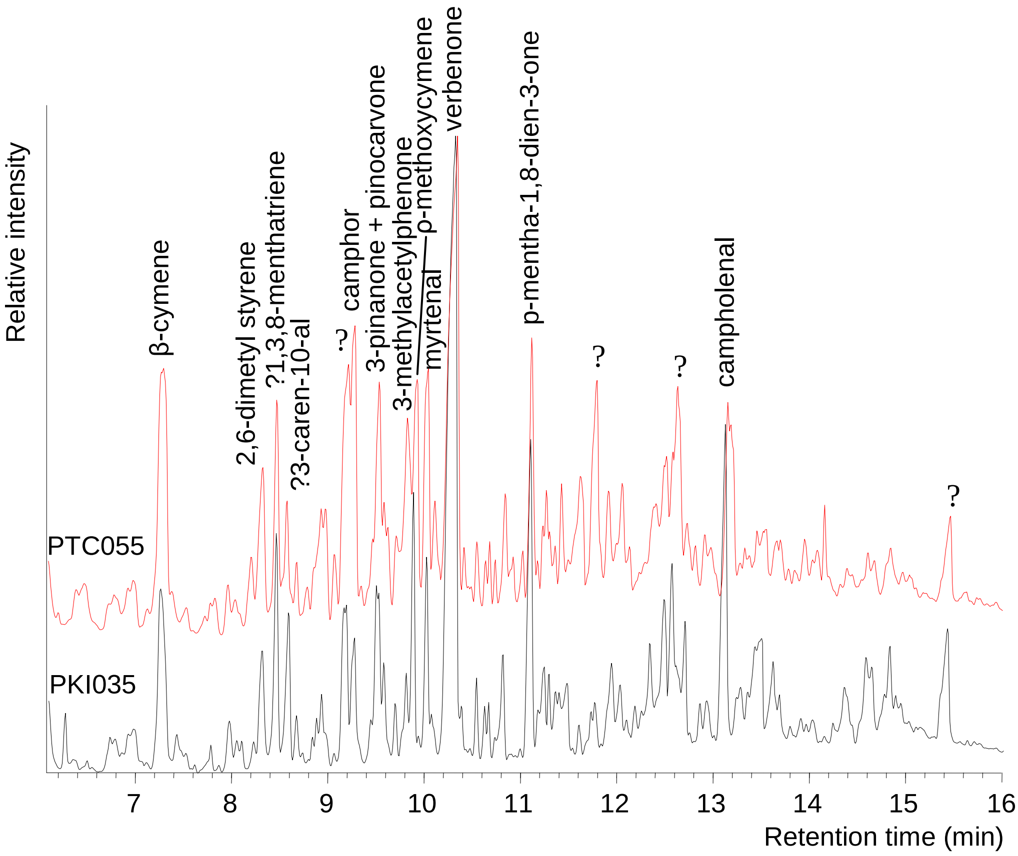
The fragments of resinous appearance from the foot area of the mummified ibis, Oriental Museum, Durham University (162/1 and 162/2) were found to contain a range of near-identical terpenoids. The LMM components are predominantly monoterpenes (**Figure 6**). These moieties are non-diagnostic as they are common to many plant exudates while their presence/absence and relative abundance varies considerably due to growing conditions, maturation stage of the plant or harvesting methods (Assimopoulou and Papageorgiou 2005a; Flamini *et al.* 2004; Langenheim 2003: 385-387). Fortunately, HMM components are diagnostic, at least to the level of genus. In this instance, both pentacyclic and tetracyclic terpenic compounds were identified (**Figure 7**). No triterpenoid acids survived although a range of biosynthetic transformation and oxidation products were present. The majority have oleanane skeletons with lupane and dammarane derivatives, including ocotillones (oxidised dammaranes), as minor contributors. No ursane related components were identified. This combination is consistent with aged *Pistacia* spp. exudates (Assimopoulou and Papageorgiou 2005a; Stern *et al.* 2003). The fragmentation patterns of the oleananes indicate that most had a Δ12 or 28-nor-Δ17 configuration although olean-18-en-3-one was also observed which suggests that the source is unlikely to be *Pistacia lentiscus* (Assimopoulou and Papageorgiou 2005b).

These observations were confirmed through comparison with modern reference resins from a variety of *Pistacia* spp. The solvent extracts obtained from the taxonomically closely related *Pistacia khinjuk* (PKI035) and *Pistacia terebinthus* (PTC055) (c.f. Golan-Goldhirsh *et al.* 2004) in particular showed a remarkable correspondence with those from the archaeological materials in both the sequence of monoterpenes (**Figure 8**) and the triterpenes (**Figure 9**) present. These samples had been curated in the reference collection at the University of Bradford for many years and so can be considered to be naturally aged although inherent variability may also be a factor. Those from the taxonomically more distinct *Pistacia lentiscus* (c.f. Golan-Goldhirsh *et al.* 2004) contained far fewer monoterpenes and were generally dominated by the classic *Pistacia* spp. biomarkers: moronic, oleanonic, *iso*masticadienonic and masticadienonic acids.



TIC (26-32 min): mummified ibis, MM13 & MM15, Oriental Museum, Durham, 162

Figure 7. Partial TIC, triterpenes in samples from mummified hawk, AEABB 162



TIC (6-16 min): modern reference *Pistacia* spp. resins

Figure 8. Partial TIC, sesquiterpenes in modern *Pistacia* spp. resins

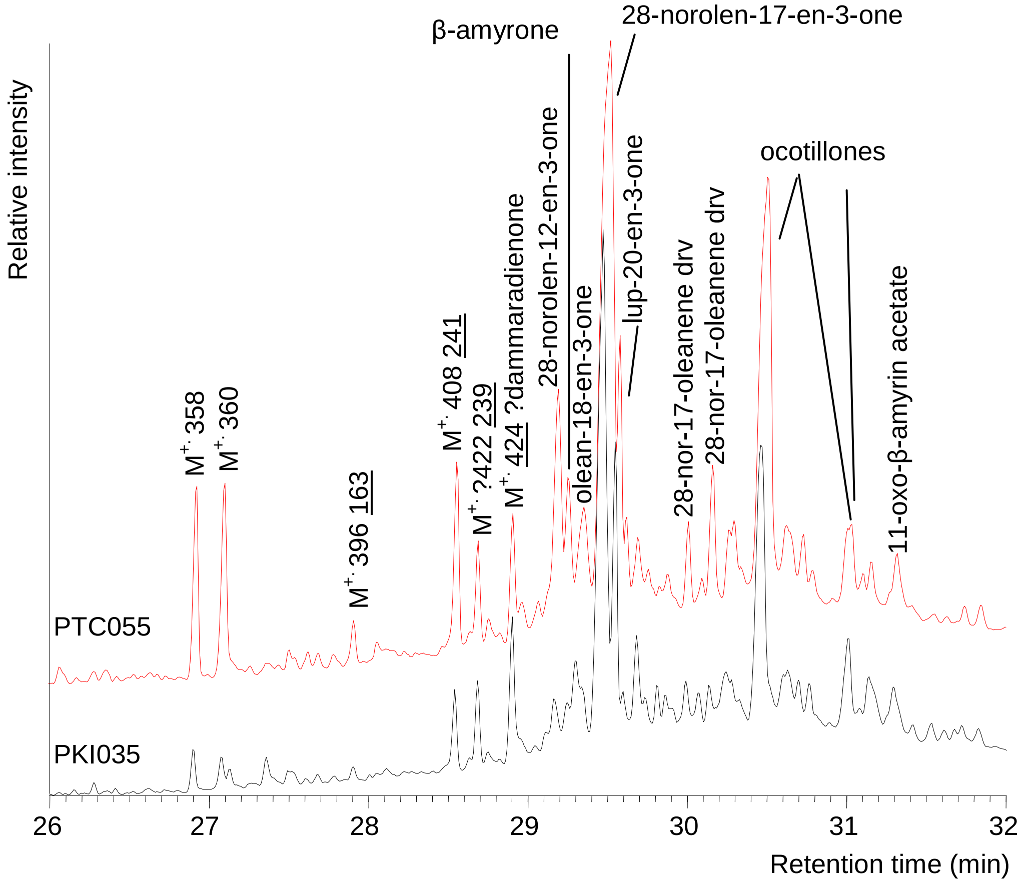
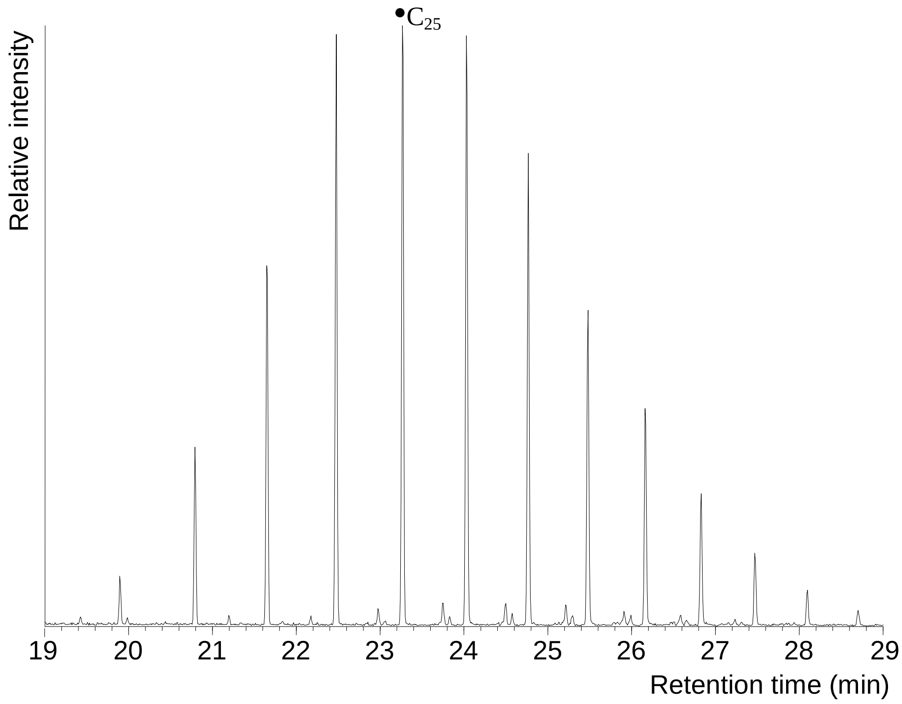


Figure 9. Partial TIC, triterpenes in modern *Pistacia* spp. resins

Complex mixtures

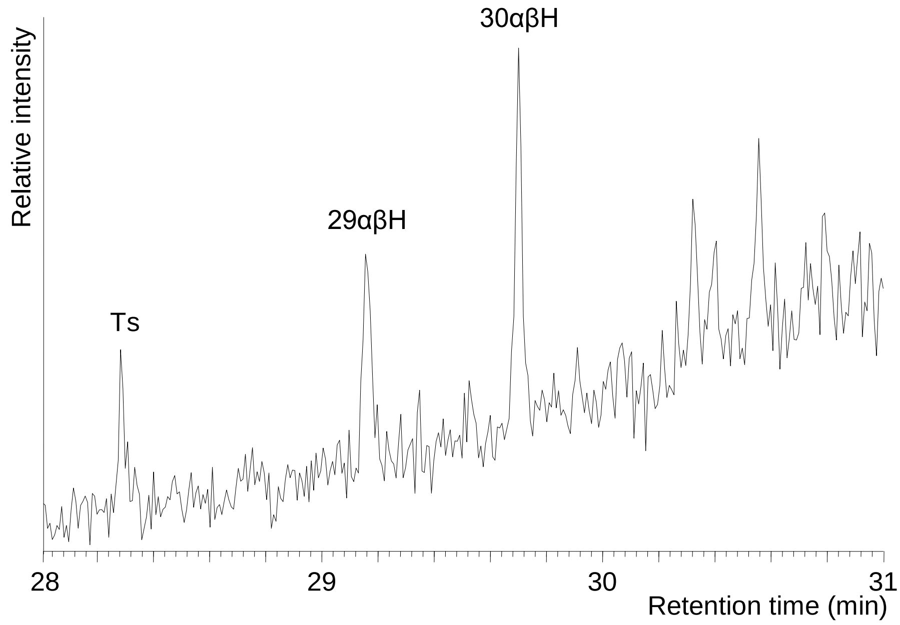
a. MM6, ibis, Elgin Museum, 471/5

The sample from the Elgin Museum mummified ibis (417/5; MM6) contained a complex mixture of components dominated by an homologous series of *n*-alkanes (C_{21-33}, C_{25} max; **Figure 10**). As traces of hopanes were observed (**Figure 11**), it is possible that bitumen derived from a coal-sourced paraffinic oil was present (Hunt 1996: 397-411; Killops and Killops 2005: 149-153, 197-203). The various LMM aromatic, sesquiterpenic and diterpenic compounds present could, theoretically, also derive from such a source (**Figure 12**). However, the range of lipid species in conjunction with an abundance of SFAs dominated by hexadecanoic acid ($C_{16:0}$) are more reminiscent of a deliberate mixture of plant products, probably an oil and a resin. Indeed, a very similar combination has been reported in a cat mummy from Beni Hassan (Buckley *et al.* 2004) and in embalming materials from human mummies (Buckley and Evershed 2001; Łucejko *et al.* 2012; Abdel-Maksoud and El-Amin 2011).



XIC (*m/z* 85): sample MM6, mummified ibis, Elgin Museum, 471/5

Figure 10. Partial XIC, *n*-alkanes from mummified ibis, AEABB 471/5

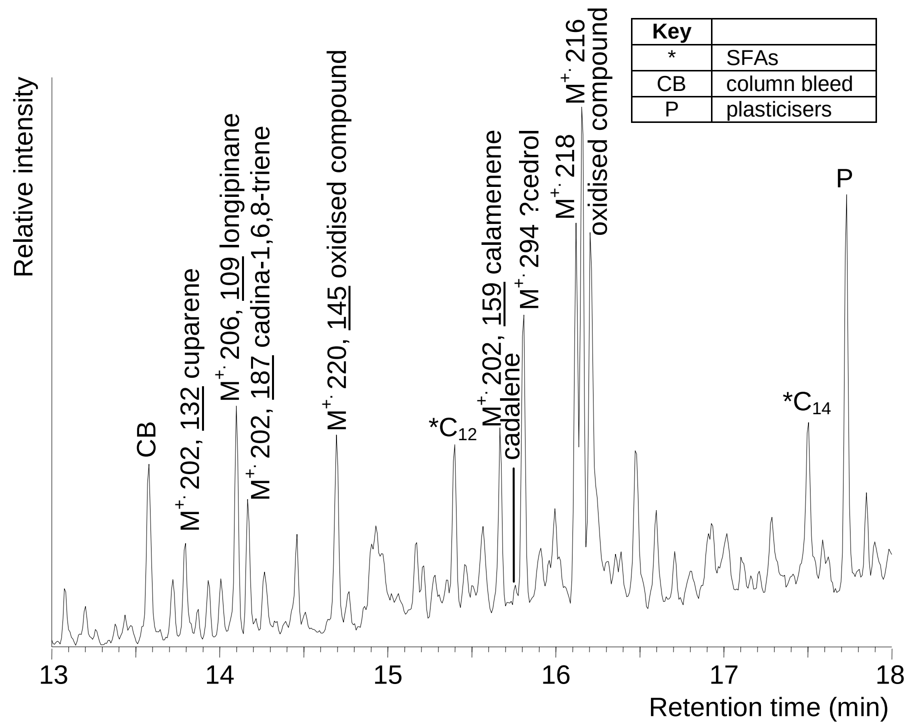


XIC (*m/z* 191): sample MM6, mummified ibis, Elgin Museum, 471/5

Figure 11. Partial XIC, hopanes from mummified ibis, AEABB 471/5

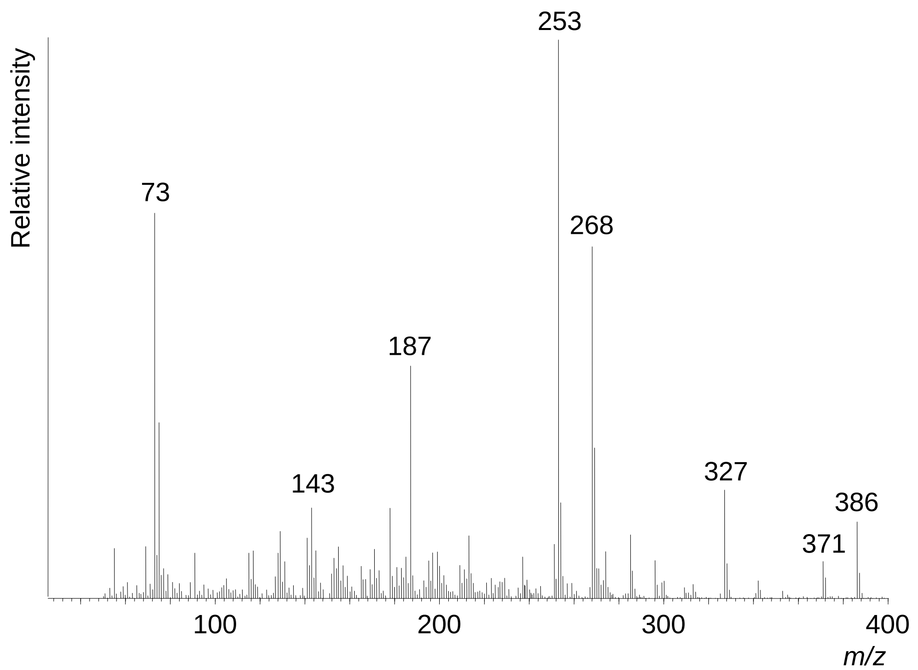
Due to their low abundance and the similar fragmentation patterns of many of the LMM aromatics and sesquiterpenes definitive identification proved problematic. The compounds clearly characterised included cuparene and calamenene (cadina-1,3,5-triene). These have been reported in the resinous wood and exudates of members of the Cupressaceae (Enzell and Erdtman

1958; Kamatou *et al.* 2010) and Pinaceae (Colombini *et al.* 2000; Koller *et al.* 2003). Tentative classification of other moieties together with two potential benzocycloheptenes ($M^{+\cdot}$ 216 and 218) as described by Buckley *et al.* (2004) appear to support the presence of a coniferous product (Adams 1987; Koller *et al*. 2003; Langenheim 2003: 329-331).



TIC (13-18 min): sample MM6, mummified ibis, Elgin Museum, 471/5

Figure 12. Partial TIC, sesquiterpenes from mummified ibis, AEABB 471/5

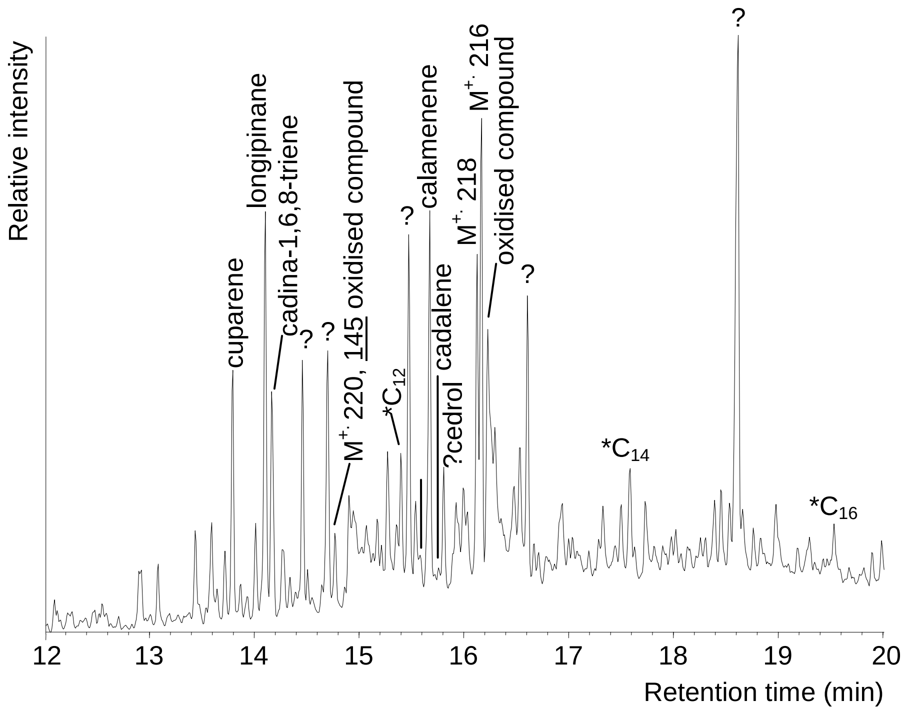


Mass spectrum (24.2 min): 7-oxodehydroabietic acid, mummified ibis, 471/5

Figure 13. Mass spectrum, 7-oxodehydroabietic acid from mummified ibis, AEABB 471/5

Moreover, the oxidised and dehydrogenated abietane-skeleton diterpenes present (e.g. 7-oxodehydroabietic acid; **Figure 13**) are characteristic biomarkers for conifer resins from the Pinaceae family which includes pines, cedars and larches (Colombini and Modugno 2009; Langenheim 2003: 37, 54-59). Although *Abies* spp. (true firs) and *Larix* spp. (larches) incorporate labdane compounds which help distinguish them (Mills and White 1999: 101) while an abundance of abietic and pimaric acids are more characteristic of *Pinus* spp. exudates (Mills and White 1977; Colombini *et al.* 2000), the diterpenoids produced by all genera are very similar (Otto *et al.* 1997). So, in the archaeological record classification is best restricted to the level of family (i.e. Pinaceae) due to the homogenising effects of degradation pathways. It appears, therefore, that this ibis was mummified using a mixture of plant-derived materials: a Pinaceae resin, a plant (conifer?) oil and a fossil hydrocarbon.

b. MM12, ibis, Oriental Museum, Durham University, 165/2

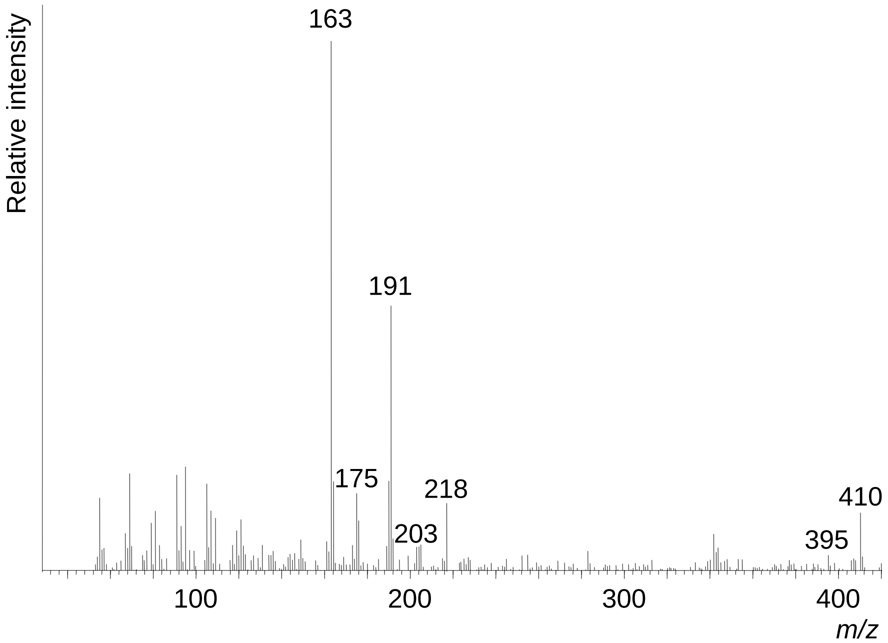


TIC (12-20 min): sample MM12, mummified ibis, Durham 165/2

Figure 14. Partial TIC, sesquiterpenes from mummified ibis, AEABB 165/2

The sample (MM12) from the mummified ibis, Oriental Museum, Durham University (165/2) appeared to consist of reeds/wood shavings and contained an homologous series of *n*-alkanes, SFAs, traces of hopanes (Ts; 29αβH;

30αβH), LMM aromatic compounds and terpenic moieties. The only diterpenoid clearly identifiable was an oxidised abietic acid derivative, methyl 7-oxodehydroabietic acid, while the LMM aromatics and sesquiterpenes (**Figure 14**) closely resembled those found in MM6. In addition, the triterpene, 28-norolean-17-en-3-one was noted (**Figure 15**). This neutral compound is thought to be formed from oleanonic acid (Pastorova *et al.* 1998) which is found in certain angiosperms such as *Pistacia* spp. and *Liquidambar orientalis* (Assimopoulou and Papageorgiou 2005a; Hovaneissian *et al*. 2008). A degradation pathway from the more widely occurring 3β-oleanolic and/or 3α-*epi*oleanolic acids also seems feasible but does not appear to have been investigated.

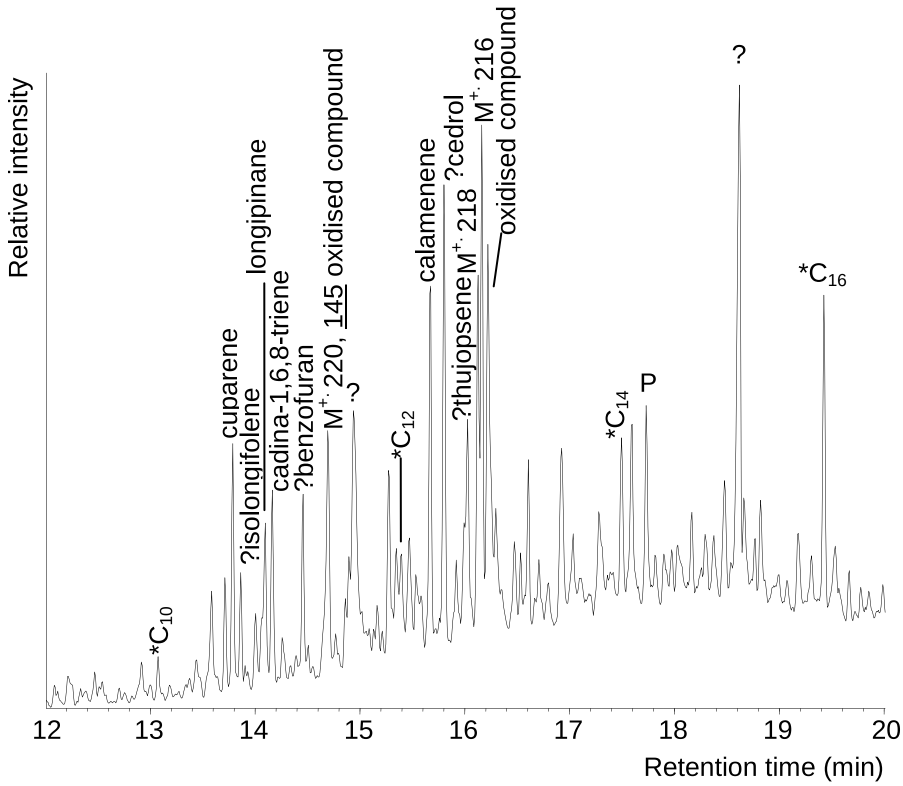


Mass spectrum (29.3 min): 28-norolean-17-en-3-one, MM12

Figure 15. Mass spectrum, 28-norolean-17-en-3-one from mummified ibis, AEABB 165/2

In this case, a mixture of plant products is again indicated with contributions from a fossil hydrocarbon and the decomposition of the body as denoted by cholesterol derivatives. Given the non-diagnostic nature of the compounds identified together with the similarities to samples MM6, MM16 and previous findings (Buckley and Evershed 2001; Buckley *et al.* 2004; Koller *et al.* 2003), it would be interesting to see if the botanical source of the reeds and other plant matter could be determined. Solvent extracts of modern reference samples could then be compared with the data presented here to see if the packing materials themselves are the source of this array of compounds as the use of *Cedrus* spp. 'sawdust' has previously been recorded (Amoros and Vozenin-Serra 1998). The potential for colonisation of the body by hopane-producing bacteria (Greenwood *et al.* 2006) which could mimic fossil hydrocarbon components might also be worth investigating. To my knowledge, this issue has not previously been considered as a confounding variable with the potential to influence the molecular composition of mummified remains.

c. MM16, animal mummy, Boston, 401/4



TIC (12-20 min): sample MM16, animal mummy, Boston 401/4

Figure 16. Partial TIC, sesquiterpenes from mummified animal, AEABB 401/4

The sample collected from the unidentified species from the Museum of Fine Arts, Boston (401/4) was found to contain an homologous series of saturated carboxylic acids (EOP, $C_{10:0\text{-}18:0}$) dominated by hexadecanoic acid ($C_{16:0}$) in conjunction with a similar range of LMM aromatics and sesquiterpenes to those observed in MM6 and MM12 (**Figure 16**). In addition, traces of triterpenic compounds were observed. Based on their key fragment ions, the majority appeared to have oleana(e)ne skeletons with 28-norolean-17-en-3-

one most abundant. Lupa(e)ne derivatives and ocotillones (oxidised dammaranes) were also present. These defunctionalised and oxidised species have been identified in degraded *Pistacia* spp. resins (van der Doelen *et al.* 1998; Stern *et al.* 2003) although the absence of diagnostic biomarker compounds means that botanical source cannot be definitively assigned. This animal mummy had, therefore, also been treated with a mixture of plant products, possibly a conifer-derived oil and triterpenoid (?*Pistacia* spp.) resin.

d. MM17, human mummy, Perth Museum and Art Gallery, Scotland, PM1

The sample of loose debris (PM1) from within the coffin of this individual contained a variety of lipid species. These consisted of an homologous series of *n*-alkanes (OEP, C_{25-33}, C_{27} max) in conjunction with palmitic wax monoesters (EOP, C_{40-44}, C_{40} max.) suggesting a contribution from epicuticular plant matter or perhaps beeswax (see 'Oils, fats and waxes' above). In addition, saturated (EOP, $C_{8:0-18:0}$; bimodal, $C_{9:0}$/$C_{16:0}$ max.), branched chain ($C_{15:0-16:0}$), monounsaturated ($C_{16:1}$ and $C_{18:1}$, cis and trans) and α,ω-di- (OEP, $C_{7:0-11:0}$; $C_{9:0}$ max.) carboxylic acids were identified. This combination suggests the presence of mammalian fat possibly of ruminant origin (Evershed *et al.* 1997) with the dicarboxylic acids indicative of thermal transformation as a result of anthropogenic heating or burial in an arid environment (Evershed 2008; Regert *et al.* 1998). LMM aromatics and triterpenic compounds were also identified with 28-norolean-17-en-3-one most abundant denoting the use of a triterpenoid resin. In addition, traces of what may be polysaccharide derivatives were detected. Sugars are of widespread occurrence in nature but in this context could denote a plant gum/gum-resin or support the presence of beeswax. As the method used here to access lipid moieties is not well-suited to the recovery and analysis of sugar components, a different approach is recommended (Bonaduce *et al.* 2007; Colombini *et al.* 2002) to confirm this observation. Thus, the human mummy curated at Perth Museum and Art Gallery, Scotland appears to have been treated with a mixture of a ruminant fat, plant wax (or beeswax), angiosperm resin and possibly a gum or gum-resin.

Conclusion

Representative samples of the debris associated with thirteen votive animal mummies and one human mummy were analysed by GC-MS. Definitive identification of many of the compounds present was limited by the low abundance and highly degraded nature of the materials available. Nonetheless, the results permitted all four objectives to be met as:

- lipids were present in most samples with the exception of the charred plant matter (MM14) and silicate fragments (MM9) although many were in such low abundance as to fall below the level of significance;
- those identified belonged to a wide range of classes from simple aliphatic hydrocarbons to wax esters and pentacyclic triterpenes;
- the nature, distribution and relative abundances of these moieties indicated that a minor contribution was present from the decomposition of the body and/or an applied animal fat in six samples (MM1, MM2, MM7, MM12, MM16, MM17) but that plant products were more widely employed both as packing materials (reeds, woody materials, charred fragments) and as part of the embalming process (plant oils, waxes and resinous exudates) with possible addition of beeswax (MM2, MM17) and, perhaps, bitumen (MM6);
- the sticky black substance (MM11) is fresh bitumen and represents a modern consolidant not an ancient treatment;
- the data indicated that ~0.05 g is sufficient if a pure lipid-rich substance is sampled but that ~0.5 g is advisable when investigating organic components within mixed debris or adhering to degraded textile wrappings.

What is of considerable interest, is that the array of compounds present is very similar to those reported in previous analyses of Egyptian mummies as summarised by Abdel-Maksoud and El-Amin (2011) although it should be noted that they refer to myrrh in error for frankincense in section 3.1.4.

Thus, through the analysis of samples of this nature when they become available, it should be possible to compile a database enabling more complex questions to be considered such as:

- did changes occur over time in the selection of materials employed in the embalming votive animal mummies?
- what relationship, if any, can be observed between the substances used in the treatment of human and animal mummies?
- were particular substances or mixtures applied to certain species?
- how does this data relate to other aspects of Egyptian ritual practices such as the significance of different species, colours and places?

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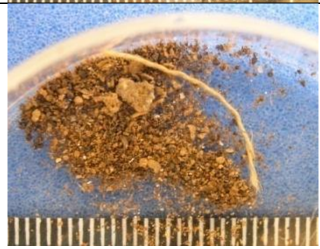
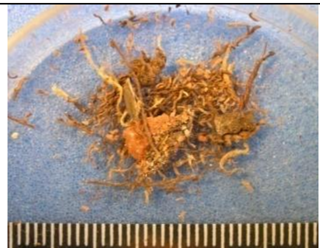
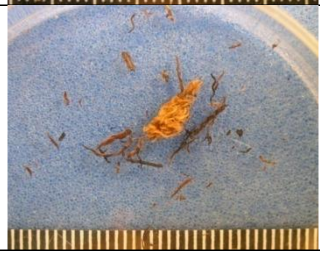
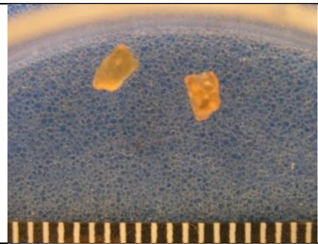
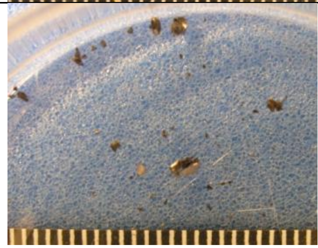
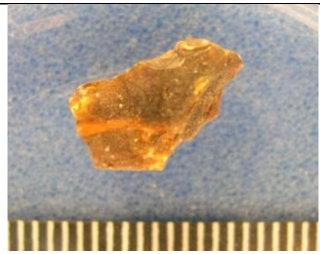
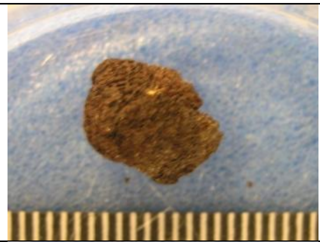
Votives, Appendix 5.1.1. Sample details and results. NB: low levels of plasticisers were present in all of the samples

Context	Samples	Sample description	Image	Mass (g)	GC-MS results
Cat 595/1 Old Speech Room Gallery, Harrow	MM1	Dark brown-black stained textile Debris from neck region - loose sweepings		0.05	**Animal fat/plant oil** ***n*-alkanes:** C_{13-33} (odd> even, C_{27} max) **SFAs:** $C_{8:0-18:0}$ (bimodal $C_{9:0}$ & $C_{14:0}$ max) **LMM aromatics (traces)**: benzoic acid ; 2-methylbenzoic acid; benzenepropanoic acid; related compounds
Cat 191/1 Bristol Museum and Art Gallery H3065	MM2	Threads of degraded textile + dark debris Linen & debris from head – loose sweepings		0.06	**Animal fat + plant oil/wax (or ?beeswax)** ***n*-alkanes:** C_{15-29} (odd>even; C_{27} max) ***n*-alkanols:** C_{18} **SFAs:** $C_{8:0-18:0}$ (bimodal $C_{9:0}$ & $C_{14:0}$ max) **Steroids:** cholesterol; cholesta-3,5-dien-7-one; cholest-4-en-3-one; cholesta-4,6-dien-3-one; cholestane-3,6-dione **Wax esters:** C_{36-42} (evens>odds)
Bird 464/2 Buckinghamshire Museum AYBCM:2003-91.2	MM3	Orange-brown debris + degraded textile Debris from head end - loose in wrappings		0.07	**Below level of significance**
Ibis 494/2 Plymouth City Museum 1920.88	MM4	Orange-brown debris + degraded textile Debris - loose in tissue paper		0.01	**Below level of significance**

Bird 465/4 Buckinghamshire Museum AYBCM:2003-91.1	MM5	Brown-black stained textile fragments + debris Linen & debris - loose under abdomen		0.03	**Below level of significance**
Ibis 471/5 Elgin Museum	MM6	Black debris + orange flecks Debris from base - loose after photography		0.02	***Pinaceae* resin + ?plant oil + fossil hydrocarbon** ***n*-alkanes:** C_{21-33} **SFAs:** $C_{8:0-18:0}$ (bimodal $C_{9:0}$ & $C_{16:0}$ dominant) **Sesquiterpenes:** cuparene; longipinane; cadina-1,6,8-triene; calamenene; cadalene; ?cedrol & oxidised compounds **Diterpenes**: didehydroabietic acid; dehydroabietic acid; methyl dehydroabietate; 7-oxodehydroabietic acid; methyl 7-oxo-dehydroabietic acid **Hopanes:** Ts; 29αβH; 30αβH
Cat 144/3 Nottingham Museum NCM 1933-281/1	MM7	Orange-brown debris + degraded textile Debris - loose in box		0.08	**Animal fat + ?plant oil** ***n*-alkanes:** C_{23-31}(C_{27} max) **SFAs:** $C_{8:0-18:0}$ (bimodal $C_{9:0}$ & $C_{14:0}$ dominant) **Steroids:** cholesterol; cholesta-3,5-dien-7-one; cholesta-4,6-dien-3-one + ?related compounds **LMM aromatics (traces):** benzoic acid; 2-methylbenzoic acid; 3-hydroxybenzoic acid
Jackal 001/1 Grantham Museum LCNGR1995.E.657	MM8	Dark brown matter, stained textile + debris Linen & debris - loose sweepings		0.04	**Below level of significance**

Cat 150/5 Derby Museum DBYMU1929-189/1	MM9	Very hard yellow-orange translucent material Translucent fragments x2 - from bag of debris		0.01	**Inorganic - possibly a silicate**
	MM10	Pale brown textile fragments Linen fragments - from bag of debris		0.02	**Below level of significance**
Hawk 146/2 Nottingham Museum HH-X 1095	MM11	Tiny pieces of black glossy material ?Resin from rear aspect - area of damage		0.005	**Fossil hydrocarbon (MODERN)** ***n*-alkanes**: C_{22-32} **Polyaromatic hydrocarbons**: phenanthrene; anthracene; fluoranthene; pyrene; 2-methyfluroanthrene; naphthacene; benzanthracene; dibenzofluorene isomers; indenopyrene isomers **Hopanes**: Ts; 29αβH; 30αβH
Ibis 165/2 Oriental Museum, Durham University DUROM.1971.122 Saqqara tomb 3508	MM12	Mixed debris including puparia, ?bone, ?reeds Debris - removed from packaging		0.1	**Plant materials + animal fat** ***n*-alkanes:** C_{21-29} (C_{23} max) **SFAs (traces):** $C_{8:0-18:0}$ ($C_{12:0}$ max) **Steroids:** cholesta-3,5,7-triene; cholestan-3-one **LMM aromatics:** benzofurans + naphthalenes **Sesquiterpenes:** cuparene; longipinane; cadina-1,6,8-triene; calamenene; cadalene; ?cedrol & oxidised compounds **?Terpene derivatives:** $M^{+\cdot}$ 358; 360; 396 **Diterpenes**: methyl 7-oxodehydroabietic acid **Triterpenes**: 28-norolean-17-en-3-one **Hopanes**: Ts; 29αβH; 30αβH

Ibis 162/2 Oriental Museum, Durham University EG727	MM13	Glossy brown/orange resinous fragment Dark-orange resin fragment from foot area		0.05	***Pistacia* spp. resin** **Monoterpenes:** β-cymene, dimethylstyrene, *p*-menthatriene, camphor, 3-pinanone, pinocarvone, 3-methylphenone, myrtenal, verbenone, *p*-mentha-1,8-dien-3-one, campholenal + various unidentified **Cyclic compounds:** bp 108 ?furans **Sesquiterpenes:** cuparene; calamenene **?Terpene derivatives:** $M^{+\cdot}$ 358; 360; 396 **Triterpenes:** lupenone; dammardienone; 28-norolean-12-en-3-one; β-amyrenone; olean-18-en-3-one; 28-norolean-17-en-3-one; lup-20-en-3-one; olean-12-ene & 28-nor-17-oleanene derivatives; ocotillones (oxidised dammaranes)
	MM14	Black, opaque mass with ?pattern/structure Piece of dark ?charred material from foot area		0.05	**Carbonised material - no lipids present**
Ibis 162/1 Oriental Museum, Durham University EG727	MM15	Two glossy orange resinous fragments Amber-coloured resin fragments from foot area		0.05	***Pistacia* spp. resin** **Monoterpenes:** β-cymene, dimethylstyrene, *p*-menthatriene, camphor, 3-pinanone, pinocarvone, 3-methylphenone, myrtenal, verbenone, *p*-mentha-1,8-dien-3-one, campholenal + various unidentified **Cyclic compounds:** bp 108 ?furans **Sesquiterpenes:** cuparene; calamenene **Terpene derivatives:** $M^{+\cdot}$ 358; 360; 396 **Triterpenes:** lupenone; dammardienone; 28-norolean-12-en-3-one; β-amyrenone; olean-18-en-3-one; 28-norolean-17-en-3-one; lup-20-en-3-one; olean-12-ene & 28-nor-17-oleanene derivatives; ocotillones (oxidised dammaranes)

Animal 401/4 Museum of Fine Arts, Boston	MM16	Dark brown-black stained mass of layered textiles Dark material with feather and textile impressions		0.25	**Triterpenoid resin, ?plant oil + animal fat** **SFAs**$_{:10:0\text{-}18:0}$ (evens>odds; $C_{16:0}$ dominant) **Steroids:** cholesta-3,5-dien-7-one **LMM aromatics:** ?benzofurans + naphthalenes **Sesquiterpenes:** cuparene;?isolongifolene; longipinane; cadina-1,6,8-triene; calamenene; cadalene; ?cedrol; ?thujopsene; oxidised compounds **?Terpene derivatives:** $M^{+\cdot}$ 358; 360; 396 **Triterpenes**: oleandienone; 28-norolean-12-en-3-one; 28-norolean-17-en-3-one; lup-20-en-3-one; ocotillones
PM1 Perth Museum & Art Gallery, Scotland	MM17	Brown stained textiles + debris Debris - loose in trough of coffin		0.1	**Animal fat, ?plant wax/beeswax, triterpenoid resin + ?gum/gum-resin** ***n*-alkanes:** $C_{25\text{-}33}$ **SFAs**: C $_{8:0\text{-}18:0}$ (evens>odds, bimodal, $C_{9:0}$/$C_{16:0}$ max.) **Branched**: $C_{15:0\text{-}16:0}$; **MUFAs**$_{:16:1 + 18:1}$ (cis + trans); **α,ω-dioic acids:** $C_{7:0\text{-}11:0}$ (odds>evens, $C_{9:0}$ max.) **LMM aromatics (?traces)**: naphthalene; benzoic acid; 2-methylbenzoic acid + ?sugar derivatives **Wax esters:** $C_{40\text{-}44}$ (evens>odds) **Terpenic compounds**: bp 163, $M^{+\cdot}$396; ?28-norolean-12-en-3-one (trace); 28-norolean-17-en-3-one; bp 163, $M^{+\cdot}$ 440

Total ion current (TIC) chromatograms of the silylated samples can be found on **Disc 1, File 5.1**.

5.2 Pilot study: analysis of materials used in the embalming of Egyptian votive mummies.

Prepared for Dr L. McKnight and Dr S. Atherton, Ancient Egyptian Bio Bank Project, University of Manchester

Background

Many Egyptian animal mummies are curated in museums across the UK. Little is known about the processes used to prepare these votive offerings as minimal textual evidence has been discovered and chemical analyses rarely undertaken. Textile fragments impregnated with residues and substances adhering to feathers from avian mummies have been observed visually and by light microscopy (Atherton 2012). Portions of these materials, detached as a result of natural wear and tear, were collected for molecular analysis by gas chromatography-mass spectrometry in order to establish a protocol for illuminating questions regarding embalming techniques. Thus, samples from six votive avian mummies were submitted for lipid analysis at the University of Bradford (**Table 1**). These materials had been stored in aluminium foil or glass vials at the University of Manchester, although their previous history is not fully known. Two modern untreated feather controls from birds of prey were also analysed. The aim of this pilot investigation was:

- to ascertain if any lipids survive within samples of this nature;
- to determine the minimum sample size required for meaningful results to be obtained;
- to assess the impact of solvent extraction on substrate i.e. linen or feather integrity;
- to identify the lipids present;
- to establish the probable source(s) of these lipids.

Method

Sample preparation

The materials were photographed and details recorded (**Appendix 5.2.1**). A sub-sample of each was solvent extracted in dichloromethane:methanol (DCM:MeOH, 2:1, *v/v*, 3 x 2 ml) aided by ultrasonication. The solvent-soluble fractions were combined and excess solvent evaporated under a stream of nitrogen. To promote separation, silyl derivatives were produced through trimethylsilylation of each dry residue using ~0.5 mL of *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% TMCS (40 °C, 15 min). Excess reagent was removed by evaporation at room temperature and the derivatised samples re-diluted in DCM (~0.1 ml) for analysis by gas chromatography-mass spectrometry (GC-MS). Disposable screw-topped glass vials were used throughout to minimise cross-contamination. All glass and metal wares triple-cleaned with dichloromethane prior to use.

GC-MS analysis

The analysis was carried out by combined gas chromatography-mass spectrometry using an Agilent 7890A GC system, fitted with a 30 m x 0.25 mm, 0.25 μm DB-5MS UI 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple axis mass selective detector. The splitless injector and interface were maintained at 300 °C and 280 °C respectively and the carrier gas, helium, at constant flow. The temperature of the oven was programmed to rise from 50 °C (isothermal for 2 min) to 350 °C (isothermal for 10 min) at a gradient of 10 °C per minute. The column was directly inserted into the ion source where electron impact (EI) spectra were obtained at 70 eV with full scan from *m/z* 50 to 800 amu.

Results and discussion

The results are presented as total ion current (TIC) and extracted ion current (XIC) chromatograms of the silylated solvent extracts with mass spectra of key compounds (**Figures 3-8; Disc 1, File 5.2**). Each separated component is shown as a discrete peak with the area beneath representative of its relative abundance. The components discussed in the text have been labelled. Assignments have been made through mass spectral interpretations based on the molecular mass and established fragmentation patterns of simple and complex lipids and their relative retention times.

Control samples

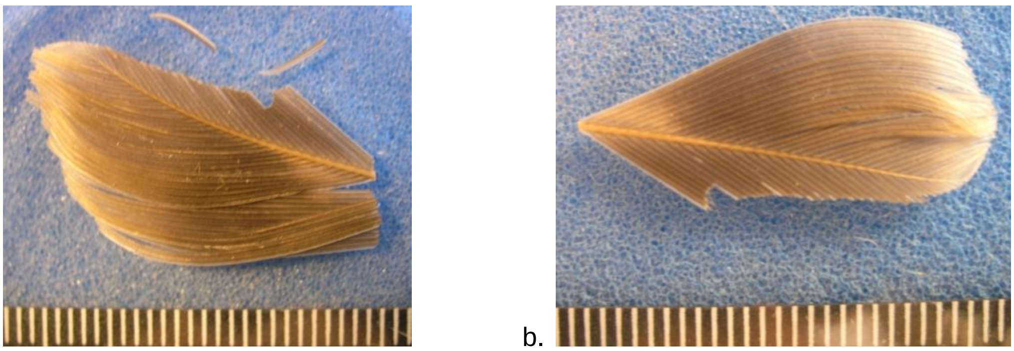
Feathers from two different identified species of birds of prey (*Accipiter nisus* and *Falco tinnunculus*) were selected as comparative reference materials.

These untreated feathers were analysed in order to ascertain the nature of any intrinsic lipids present in the fraction obtained. The lipids produced by birds has been shown to vary according to species, season, health and nutrition (Jacob 1976). In vivo, those associated with the feathers may derive from the epidermis or gland secretions (e.g. preen/uropygial) and predominantly comprise wax esters, triacylglycerols and their degradation products (e.g. alcohols, carboxylic acids) and cholesterol (Rajchard 2010).

Both sample extracts contained only traces of ubiquitous lipids. These comprised saturated carboxylic acids (SFAs, $C_{16:0}$ and $C_{18:0}$) together with cholesterol and 5α-cholestanol, its aerobic microbial reduction (biohydrogenation) product (Bull *et al.* 2002). These results provide a baseline from which to consider the archaeological samples and demonstrate that, although recognisable, these intrinsic lipids fall below the level of significance for this technique (i.e. are in lower abundance than the column bleed).

Impact of solvent extraction on substrate

The controls were also used to consider the impact of solvent extraction on the integrity of the feathers. Both remained intact and, to the naked eye, appeared visibly unaltered by the process (**Figure 1**). Microscopy should, however, be used to evaluate these findings.



a. b.

Figure 1. Modern reference feather, *Accipiter nisus*: a. before solvent extraction b. after solvent extraction

Likewise, little alteration was observed in the substrates from which the archaeological residues were obtained (**Appendix 5.2.1**). The structural details of both the feathers and, perhaps more importantly for other forms of analysis, the textiles were maintained (**Figure 2**). This is of considerable importance as it demonstrates that these materials can be returned intact to the contributing museums once the organic residues have been extracted.

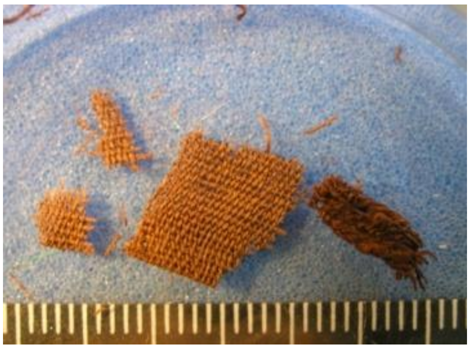
a.  b. 

Figure 2. Archaeological textile fragments, bird of prey: a. before solvent extraction b. after solvent extraction

Minimum sample size

With regards to the archaeological materials, another aspect assessed was the minimum amount of sample required for meaningful results to be obtained. As mass balances of sufficient accuracy are unlikely to be available in many contexts, the relative dimensions of the textile fragments and feathers were also considered. Previous research has shown that, where a pure, lipid-rich substance such as a resin fragment is available 0.02 g (~2 mm^3) enabled identification of source with 0.05 g (~5 mm^3) recommended for confirmatory or further analysis to be undertaken.

When the sample consisted of mixed detritus with a considerable contribution from inorganic components and/or degraded textiles, however, an order of magnitude greater (~0.5 g) was desirable although some indication as to the substance(s) present was still able to be obtained from >0.05 g. Where the residues were impregnated within or adhering to substrates of differing density, textile fragments c. 10 mm^2 (~0.05-0.1 g) and feather portions c. 20 mm x 10 mm (~0.01-0.05 g) in dimensions were found to be advisable. Thus, of the archaeological samples analysed four contained low abundance non-diagnostic lipids (MM24, 27-29) which fell below the level of the instrumental artefacts and so cannot be considered of significance (**Appendix 5.2.1**).

Table 1. Details of the provenance and nature of the Ancient Egyptian mummy samples analysed

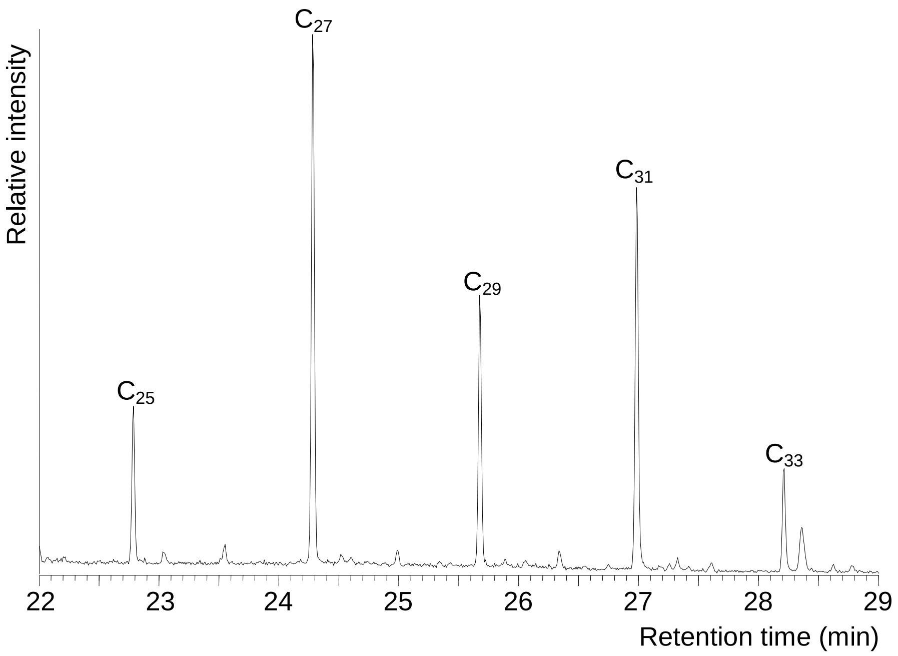
GC-MS no.	AEABB no.	Species	Sample	Mass (g)	Description	Institution details	Institution reference
MM18	004/KM/02	Bird of prey	Linen - loose on left side of bundle	0.053	Dark-stained linen	Kirklees	2003-269
MM19	004/KM/03	Bird of prey	Linen - loose on right side of bundle	0.050	Dark-stained linen		
MM20	004/KM/04	Bird of prey	Linen - loose in bag	0.102	Dark-stained linen		
MM21	004/KM/07	Bird of prey	? - loose on right side of bundle	0.018	Dark-stained material		
MM22	384/MFAB/01	*Accipiter* sp.	Feather/coating - left shoulder region	0.004	Stained feather	MFAB	Eg. Inv. 6095
MM23	384/MFAB/02	*Accipiter* sp.	Feather/coating - left wing feather, top layer	0.047	Stained feather		
MM24	384/MFAB/03	*Accipiter* sp.	Feather/coating - left wing feather, mid layer	0.012	Stained feather		
MM25	027/LV/01	*Falco* sp.	Linen - debris within tissue paper	0.054	Dark-stained linen	Garstang, Liverpool	E5408
MM26	475/JE/01	Bird of prey	Feather - exposed tail feather, inner layer	0.030	Stained feather	Leicester/New Walk	L.A100.1912.1.0
MM27	475/JE/02	Bird of prey	Feather - exposed distal end feather, outer layer	0.002	Stained feather		
MM28	153/RA/01	Bird of prey	Feather - exposed distal end	0.002	Stained feather	RAMM	106/1920/7
MM29	188/LM/02	?	?	0.003	Stained feather	?	?
MM30	EM1	*Accipiter nisus*	Feather - tail tip	0.005	Modern untreated feather	KNH, Manchester	EM1
MM31	EM2	*Falco tinnunculus*	Feather - primary tip	0.005	Modern untreated feather	KNH, Manchester	EM2

Contaminants and interferences

The majority of the samples contained low levels of phthalate plasticisers, biphenyls and related compounds. These are modern contaminants probably derived from plastic packaging which had come in contact with the mummy bundles at some point in their history. In some of the samples, a number of peaks resemble underivatised SFAs. It is possible that this is the result of trimethylsilyl (TMS) substitution having been hindered by some intrinsic factor such as interference by metal ions (?derived from the use of natron as a desiccant) with partial saponification of these lipids having prevented the TMS group from attaching effectively to the carboxyl (-COOH) functionalities. These broad fronting peaks may, however, also originate from contact with modern plastics which contain oil-derived components to add flexibility.

Complex mixtures: oils/fats, waxes and plant exudates

1. Bird of prey, Leicester, New Walk, L.A100.1912.1.0 (MM26 + MM27)



XIC (*m/z* 85): bird of prey, Leicester (MM26)

Figure 3. XIC (*m/z* 85), *n*-alkanes, bird of prey, Leicester, New Walk (MM26)

In the samples from this mummy bundle, HMM *n*-alkanes with an odd-over-even (OEP) carbon number predominance (C_{25}-C_{33}) and long-chain monoesters of palmitic acid ($C_{36-42,}$ C_{40} max) were present (**Figures 3 and 4**). These may derive from the epicuticular waxes of higher plants (Rieley *et al.*

1991) although beeswax is an alternative source as the *n*-alkane maximum falls at C_{27}. The latter substance has previously been identified in both human and animal mummies (Buckley and Evershed 2001; Buckley *et al.* 2004) but is generally characterised by wax esters greater than 40 carbons in length (C_{46} max) with their degradation products, HMM *n*-alkanols and carboxylic acids (Garnier *et al.* 2002; Heron *et al.* 1994). The latter were not observed here although it should be noted that an absence of *n*-alkanols and reduction in ester components has been reported in experimentally heated beeswax (Namdar *et al.* 2009).

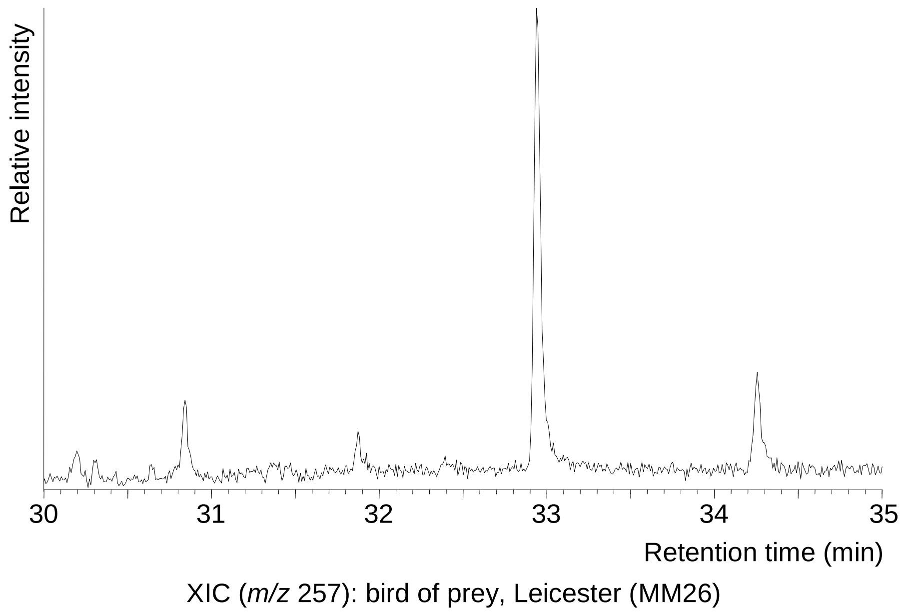


Figure 4. XIC (*m/z* 257), wax esters, bird of prey, Leicester, New Walk (MM26)

Traces of triterpenic compounds were also noted. Based on their characteristic fragmentation patterns these comprised of:

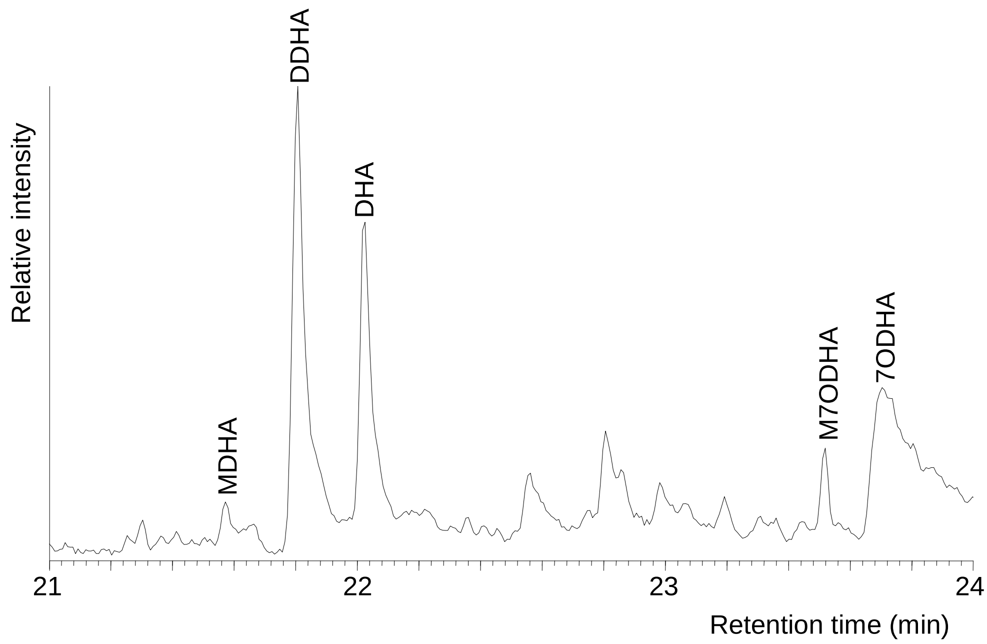
- 28-norolean-17-enes, base peak at *m/z* 163, significant fragment ion at *m/z* 191, molecular ions at *m/z* 396, 410 (28-norolean-17-en-3-one) and 440 (Budzikiewicz *et al.* 1963);
- dammarane derivatives, base peak at *m/z* 315, significant fragment ions at *m/z* 205, 163, 109, molecular ions at *m/z* 358 and ?426 (Assimopoulou and Papageorgiou 2005);
- three ocotillones (oxidised dammaranes with furan derivative side chains), base peak at *m/z* 143 (van der Doelen *et al*. 1998), significant fragment ions depend on chain length e.g. *m/z* 399 with molecular ion

at *m/z* 458 although near complete fragmentation means that the latter is rarely visible as is the case here.

This combination suggests the presence of an highly degraded and/or heated triterpenoid resin and resembles the range of compounds reported in aged *Pistacia* spp. exudates (Stern *et al.* 2003).

2. *Accipiter* sp., MFAB, Eg. Inv. 6095 (MM22-24)

A mixture of substances appears to be present in the samples from this mummy bundle. They may include some contribution from the degradation of body tissues or the application of a fat or oil, as a limited range of even carbon chain SFAs ($C_{14:0,\ 16:0,\ 18:0}$; $C_{16:0}$ dominant) were observed although these are too ubiquitous to be assigned a definitive source. The HMM *n*-alkanes with an odd-over-even (OEP) carbon number predominance (C_{27}-C_{33}) and long-chain monoesters of palmitic acid ($C_{38-46,}$ C_{40} max), may derive from the epicuticular waxes of higher plants due the *n*-alkane maximum at C_{31} (Rieley *et al.* 1991) although beeswax is again a possibility. Traces of triterpenic compounds were also identified (28-norolean-17-en-3-one, two related compounds, three ocotillones) alongside a number of abietane skeleton diterpenes (**Figure 5; Table 2**).



TIC (21-24 min): *Accipiter* sp., MFAB (MM22)

Figure 5. TIC, diterpenes, *Accipiter* sp., MFAB (MM22), identifiers relate to **Table 2**

These are end products of the degradation of ?*Pistacia* spp. and Pinaceae resinous plant exudates respectively (Colombini and Modugno 2009). The absence of any primary resin acids and presence of only abietane derivatives from the Pinaceae contribution indicate that this mixture may have been heated in antiquity (Egenberg *et al.* 2002; Stern *et al.* 2003). Natural oxidation processes can, however, produce similar results (Mills and White 1977).

Table 2. Identification of diterpenic compounds, *Accipiter* sp., MFAB (MM22), identifiers relate to **Figure 5**

Code	$M^{+\cdot}$	BP	Fragment ions	Identification
MDHA	314	239	55, 141, 239, 255, 299	methyl dehydroabietate
DDHA	370	237	73, 103, 143, 195, 209, 252, 355	didehydroabietic acid
DHA	372	239	73, 129, 143, 171/3, 185, 240, 255	dehydroabietic acid
M7ODHA	328	253	313, 296, 269, 254, 213, 187	methyl 7-oxodehydroabietate
7ODHA	386	253	73, 143, 187, 268, 327	7-oxodehydroabietic acid

3. Bird of prey, Kirklees, 2003-269 (MM18-21)

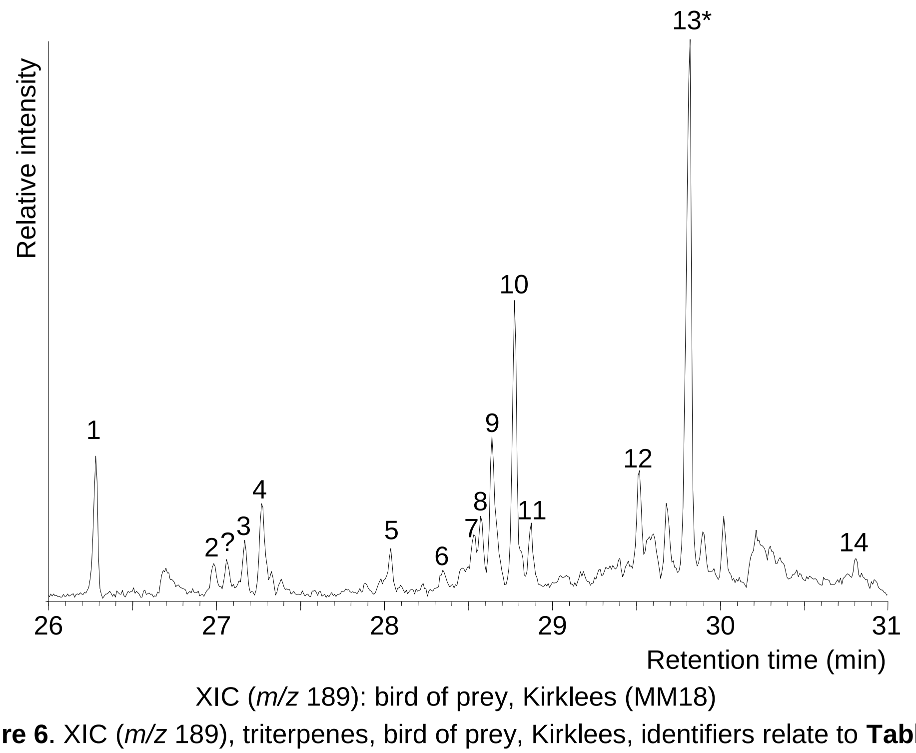


Figure 6. XIC (*m/z* 189), triterpenes, bird of prey, Kirklees, identifiers relate to **Table 3**.

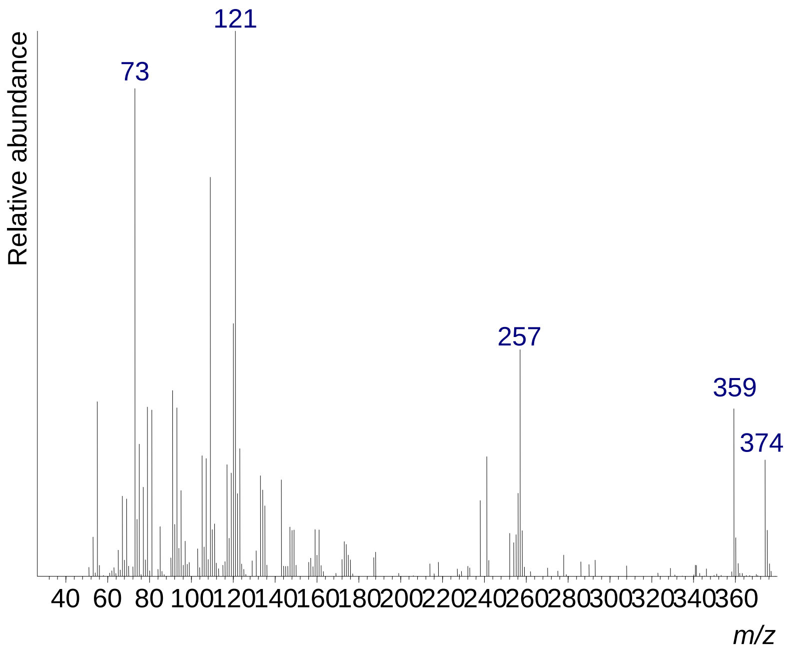
Evidence for a mixture of substances was found in the samples from this bird of prey. A limited range of even carbon chain SFAs ($C_{14:0,\ 16:0,\ 18:0}$; $C_{16:0}$ dominant) was accompanied by cholesterol and a number of its derivatives (**Appendix 5.2.1**). The latter confirm the presence of an animal fat which could be intrinsic (from the decomposition of the body) or extrinsic (applied as part of a scented unguent) in origin. Triterpenic compounds were also observed. Based on their characteristic fragmentation patterns these are neutral derivatives with predominantly oleana(e)ne skeletons alongside dammarane derivatives and their oxidised forms, ocotillones (**Figure 6; Table 3**). This combination again indicates the presence of a highly degraded triterpenoid resin and may represent an aged and/or heated *Pistacia* spp. exudate.

Table 3. Identification of triterpenes, bird of prey, Kirklees (MM18) assignments based on fragmentation patterns after Assimopoulou and Papageorgiou (2005), identifiers relate to **Figure 6**

ID	$M^{+\bullet}$	BP	Key fragments	Name of compound
1	358	95	343, 315, 297, 205, 189, 177, 163	dammarane derivative
2	394	204	381, 281, 189, 175, 135, 119	a nor-olean-12-diene
3	394	394	379, 281, 207, 189, 175, 163, 119	a nor-olean-18-diene
4	396	163	381, 203, 191, 175, 121	28-nor-17-oleanene derivative
5	422	422	407, 281, 269, 255, 239, 207, 133	?olean-18-dien-3one
6	?	143	411, 385, 367, 191, 175, 161, 125	ocotillone
7	408	408	393, 281, 269, 207, 189, 174, 161	norolean-18-ene derivative, hydroxyl at C-3
8	406	406	391, 281, 218, 207, 131, 119	norolean-18-ene derivative, hydroxyl at C-3
9	424	177	409, 281, 253, 205, 189, 121	olean-18-ene derivative, keto at C-3
10	410	163	395, 205, 191, 175, 133, 119	28-norolean-17-en-3-one
11	424	95	409, 281, 218, 205, 191, 177, 163	28-norolean-17-ene derivative
12	?	204	281, 253, 189, 175, 161	12-oleanane derivative
13	440	163	425, 215, 203, 189, 175	28-norolean-17-ene derivative, CHO at C-6
*	?	143	399, 203, 189, 175, 161, 125	ocotillone co-eluting
14	?	143	411, 203, 189, 175, 161, 125	ocotillone

In addition, diterpenic compounds were noted in sample MM18, the dark-stained linen on the left side of the bundle. Based on their characteristic fragmentation patterns, these comprise of dehydrogenation and oxidation products of abietic acid (methyl dehydroabietate, dehydroabietic acid, 7-oxodehydroabietic acid) together with pimaric acid (**Figure 7**). Compounds with abieta(e)ne and pimara(e)ne skeletons are biomarkers for coniferous resins from the Pinaceae subfamily which includes pines, firs, spruces, cedars and larches (Colombini and Modugno 2009; Langenheim 2003: 37, 54-59). Although the diterpenoid acids produced by all of these genera are very similar (Otto *et al.* 1997), an abundance of abietic and pimaric acids is considered more characteristic of *Pinus* spp. with the moieties observed here consistent with aged exudates (Colombini *et al*. 2003; Mills and White 1977).

These degraded molecular species could also be indicative of the processing of Pinaceae resins or resinous woods in antiquity (Robinson *et al.* 1987). Experimental studies have shown that thermal degradation leads to dehydrogenation, decarboxylation and a higher degree of aromatisation. Thus, as with environmental degradation, a mixture of DHA acid and various neutral compounds is produced although, as the degree of alteration is temperature dependent, relative abundances tend to be highly variable (Egenberg *et al.* 2002). In Pinaceae resin tars retene appears to be the final stable product (Robinson *et al.* 1987) whereas significant levels of methyl dehydroabietate (MDHA) may indicate the pyrolysis of resinous Pinaceae wood (Colombini *et al.* 2003). The latter compound results from the reaction between methanol, released by the heating of the wood, and the diterpenoid acids present. Sample preparation procedures (e.g. solvent extraction using DCM:methanol with warming on a hot-plate or derivatisation using methylation) can, however, increase the amount of MDHA making it an unreliable indicator (Hjulström *et al.* 2006). In this instance, therefore, heating can neither be proved nor disproved.

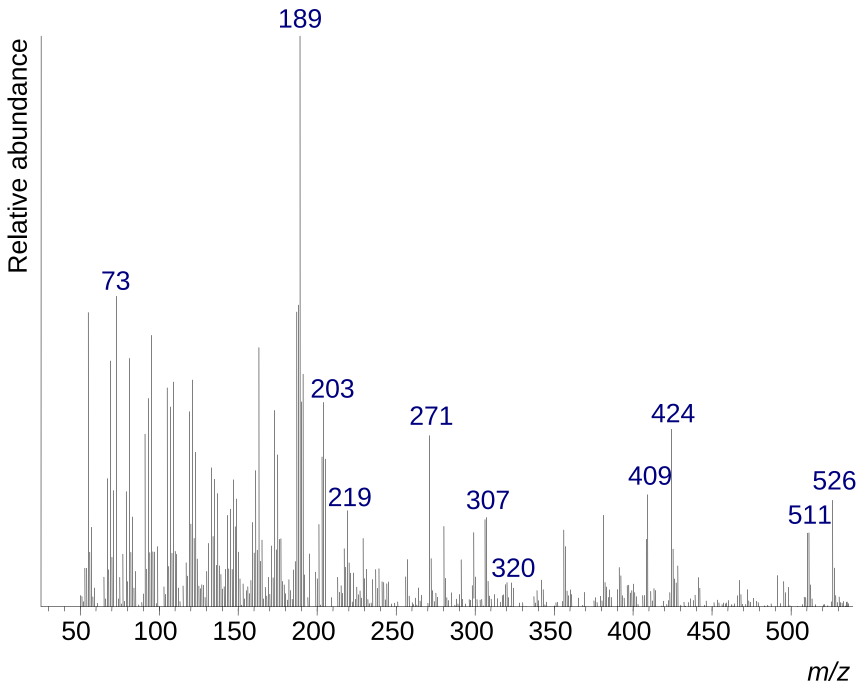


Mass spectrum (21.4 min): bird of prey, Kirklees (MM18)

Figure 7. Mass spectrum, pimaric acid, bird of prey, Kirklees (MM18)

4. *Falco* sp., Garstang, Liverpool (MM25)

A similar mixture of substances seems to have been applied to the *Falco* sp. mummy curated at Garstang, Liverpool. The presence of medium length carbon chain SFAs ($C_{12:0\text{-}20:0}$, $C_{16:0}$ max, $C_{15\text{-}17}$ isomers) may reflect a contribution from the degradation of the body tissues and/or the application of a fat/oil. The use of a natural wax is evident, however, in the combination of HMM *n*-alkanes (OEP, C_{27}-C_{33}, C_{27} max), an extensive range of long chain SFAs ($C_{22:0}$-$C_{30;0,}$ $C_{24:0}$ max), traces of *n*-alkanols (C_{18}, C_{20}) and long-chain monoesters of palmitic acid (EOP, $C_{32-46,}$ C_{40} max). This pattern most closely reflects that described for beeswax (Regert 2009) which may be supported by traces of glycerol and possibly cyclicised sugars (pyranoses/furanoses). A number of di- and triterpenic compounds were also present based on their characteristic fragmentation patterns. These include didehydroabietic acid (DDHA), methyl 7-oxodehydroabietate (M7ODHA), 28-norolean-17-en-3-one, compounds with oleana(e)ne skeletons, ocotillones (oxidised dammaranes) and what may be moronic acid (**Figure 8**). Although the remainder of these triterpenic moieties are too degraded to be clearly identifiable, it appears that they again comprise the end products of the degradation of Pinaceae and *Pistacia* spp. resins.



Mass spectrum (29.8 min): *Falco* sp., Garstang (MM25)
Figure 8. Mass spectrum, moronic acid, *Falco* sp., Garstang (MM25)

Conclusion

Textile fragments impregnated with residues and substances adhering to feathers from six votive avian mummy bundles were analysed by gas chromatography-mass spectrometry. Definitive identification of many of the compounds present was limited by the low abundance and highly degraded nature of the materials available. Nonetheless, the results permitted the objectives to be met as:

- traces of lipids were present in almost all of the samples although those from the untreated control feathers and the majority of the samples <0.01 g were in such low abundance as to fall below the level of significance;
- those identified were found to belong to a wide range of classes from simple aliphatic hydrocarbons to wax esters and pentacyclic triterpenes;
- the nature, distribution and relative abundances of these moieties indicated that a minor contribution was present from the decomposition of the body and/or an applied animal fat in five samples (MM18-21, MM25) but that plant products were more widely employed as part of the embalming process (MM18-23, MM25-26) with the possible addition of beeswax (MM23, MM25-26);
- the data indicated that ~0.05 g is generally sufficient with regards to residue-impregnated textiles (~10 mm^2) and coated feathers (~20 x 10 mm);
- visually, little damage appeared to have been inflicted upon these substrates by the solvent extraction method utilised.

What is again of considerable interest, is the range of substances identified (plant oil/animal fat, beeswax, Pinaceae and *Pistacia* spp. resins) not only in the treatment of these avian mummies but in comparison with results obtained from previous analyses of votive animal bundles (Buckley *et al.* 2004; Bruno 2013), human mummies (Buckley *et al.* 2001; Colombini *et al.* 2000; Jones *et al.* 2014) and even victual offerings (Clark *et al.* 2013). This may, in part, be due to the limitations of using a single method and so future work could include investigation of saccharide components (Colombini *et al.* 2002). Exploration of the potential of nuclear magnetic resonance might also be fruitful given the results of a blind test on an ointment containing a similar mixture to those used in embalming (Colombini *et al.* 2011). These pilot studies have, however, demonstrated the level of information that can be gleaned from this previously neglected resource. A systematic study of 'mummy dust' residues from bundles curated in museums across the UK should enable a database to be compiled which would serve to elucidate more complex questions such as:

- did changes occur over time in the selection of materials employed in the embalming votive animal mummies?
- what relationship, if any, can be observed between the substances used in the treatment of human and animal mummies?
- were particular substances or mixtures applied to certain species?
- how does this data relate to other aspects of Egyptian ritual practices such as the significance of different species, colours and places?

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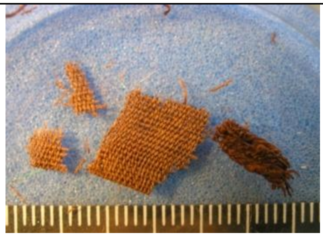
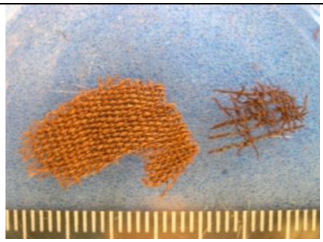
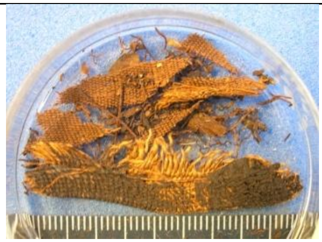
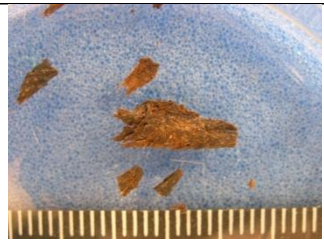
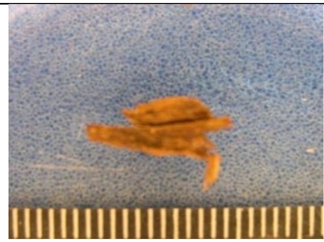
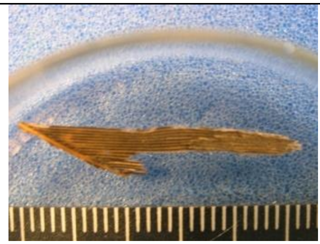
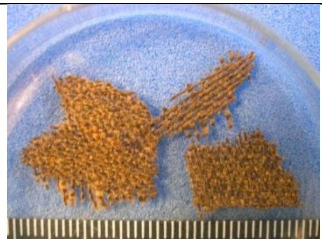
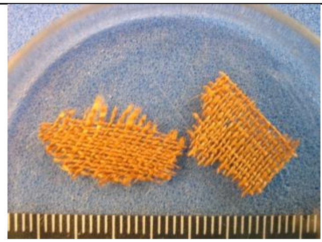
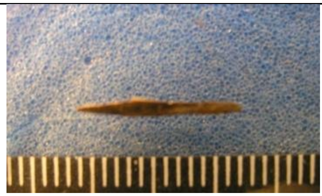
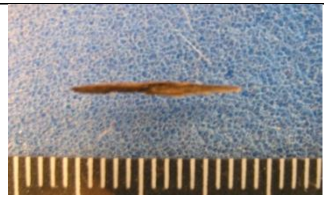
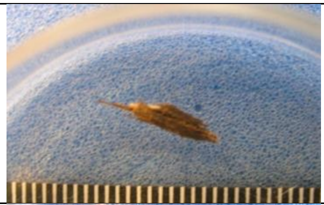
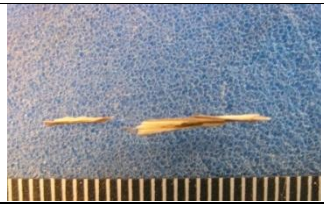
Votives, Appendix 5.2.1. Sample details and results. NB: low levels of plasticisers present in all of the samples

Context	Samples	Sample description	Image Before SE	Image After SE	Mass (g)	GC-MS results
Bird of prey Kirklees 2003-269	MM18	Dark brown residue on textile fragments Linen loose on left side of bundle			0.053	**Animal fat + plant exudates** **SFAs**: $C_{16:0}$; $C_{18:0}$ **Steroids:** cholestan-3-one; cholestan-3,5-dien-7-one; cholest-4-en-3-one **Diterpenes:** pimaric acid; methyl dehydroabietate; dehydroabietic acid; 7-oxodehydroabietic acid **Triterpenes**: oleanenes, dammaranes + ocotillones
Bird of prey Kirklees 2003-269	MM19	Dark brown residue on textile fragments Linen loose on right side of bundle			0.050	**Animal fat + plant exudates** ***n*-alkanes**: C_{23-31}, OEP **SFAs**: $C_{16:0}$; $C_{18:0}$ **Steroidal compounds:** cholestan-3-one; cholestan-3,5-dien-7-one; cholest-4-en-3-one **Triterpenes**: oleanenes, dammaranes + ocotillones
Bird of prey Kirklees 2003-269	MM20	Brown-black residue on textile fragments Linen loose in bag			0.102	**Animal fat + plant exudates** ***n*-alkanes**: C_{23-31}, OEP **SFAs**: $C_{16:0}$; $C_{18:0}$ **Steroidal compounds:** cholestan-3-one; cholestan-3,5-dien-7-one; cholest-4-en-3-one **Triterpenes**: oleanenes, dammaranes + ocotillones

Bird of prey Kirklees 2003-269	MM21	Dark brown residue on unidentified material Loose on right side of bundle			0.018	**Animal fat + plant exudates** **Steroidal compounds:** cholestan-3-one; cholestan-3,5-dien-7-one; cholest-4-en-3-one **Triterpenes**: oleanenes, dammaranes + ocotillones
Accipiter sp. MFAB Eg. Inv. 6095	MM22	Brown residue coating feather Feather from left shoulder region			0.004	**Conifer resin + ?oil/fat** ***n*-alkanes**: C_{27-33}, OEP **SFAs**: $C_{14:0;16:0;\ 18:0}$ **Diterpenes**: didehydroabietic acid; dehydroabietic acid; 7-oxodehydro-abietic acid
Accipiter sp. MFAB Eg. Inv. 6095	MM23	Brown residue coating feather Wing feather from left side, top layer			0.047	**Plant wax or beeswax** ***n*-alkanes**: C_{27-35}, OEP **SFAs**: $C_{16:0;}$ $C_{18:0}$ - traces **Wax esters:** $C_{36-44,}$ EOP **Triterpenes**: $M^{+\cdot}$ 396, 410, 440
Accipiter sp. MFAB Eg. Inv. 6095	MM24	Brown residue coating edge of feather Wing feather from left side, mid layer			0.012	**Below level of significance** ***n*-alkanes**: C_{25-33}, OEP **Triterpenes**: $M^{+\cdot}$ 396, 410, 440

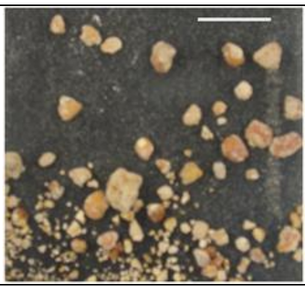
Falco sp. Garstang, Liverpool E5408	MM25	Brown residue on textile Linen fragments loose within tissue paper			0.054	**?Animal fax, beeswax + plant exudates** Benzoic + vanillic acid + derivatives Glycerol + ?cyclic sugars ***n*-alkanes**: C_{27-33}, OEP, C_{27} max ***n*-alkanols:** $C_{18:0;}$ $C_{20:0}$ **Dicarboxylic acids**: $C_{4:0-10:0}$ **SFAs**: $C_{12:0-30:0,}$ EOP ($C_{16:0}$ + $C_{24:0}$ bimodal), $C_{15:0-17:0}$ (isomers) **Wax esters:** $C_{32-46,}$ EOP, C_{40} max **Diterpenes**: didehydroabietic acid, methyl 7-oxodehydroabietate **Triterpenes**: ?moronic acid bp 163 x3 dammaranes bp 143 x3 ocotillones
Bird of prey Leicester/New Walk L.A100.1912.1.0	MM26	Brown residue coating feather Tail feather exposed, inner layer			0.030	**Plant wax/?beeswax + resin** ***n*-alkanes**: C_{23-33}, OEP **Wax esters:** $C_{36-44,}$ EOP **Triterpenes**: bp 163 + 143
Bird of prey Leicester/New Walk L.A100.1912.1.0	MM27	Brown residue coating feather fragment Feather exposed distal end, outer layer			0.002	**Below level of significance** ***n*-alkanes**: C_{25-33}, OEP **Triterpenes**: bp 163 + 143

Bird of prey RAMM 106/1920/7	MM28	Brown residue coating feather fragment Feather exposed distal end			0.002	**Below level of significance**
?	MM29	Brown residue coating ?feather fragment			0.003	**Below level of significance** ***n*-alkanes**: C_{25-33}, OEP **Triterpenes**: bp 163 $M^{+\cdot}$ 396, 410, 440
Accipiter nisus KNH, Manchester	MM30	Untreated feather Modern feather, tail tip selected as control			0.005	**Below level of significance** **Steroids**: cholesterol; 5α-cholestanol
Falco tinnunculus KNH, Manchester	MM31	Untreated feather Modern feather, primary tip selected as control			0.005	**Below level of significance** ***n*-alkanes**: C_{25-29}, OEP **SFAs**: $C_{16:0;}$ $C_{18:0}$ **Steroids**: cholesterol; 5α-cholestanol

Total ion current (TIC) chromatograms of the silylated samples can be found on **Disc 1, File 5.2**.

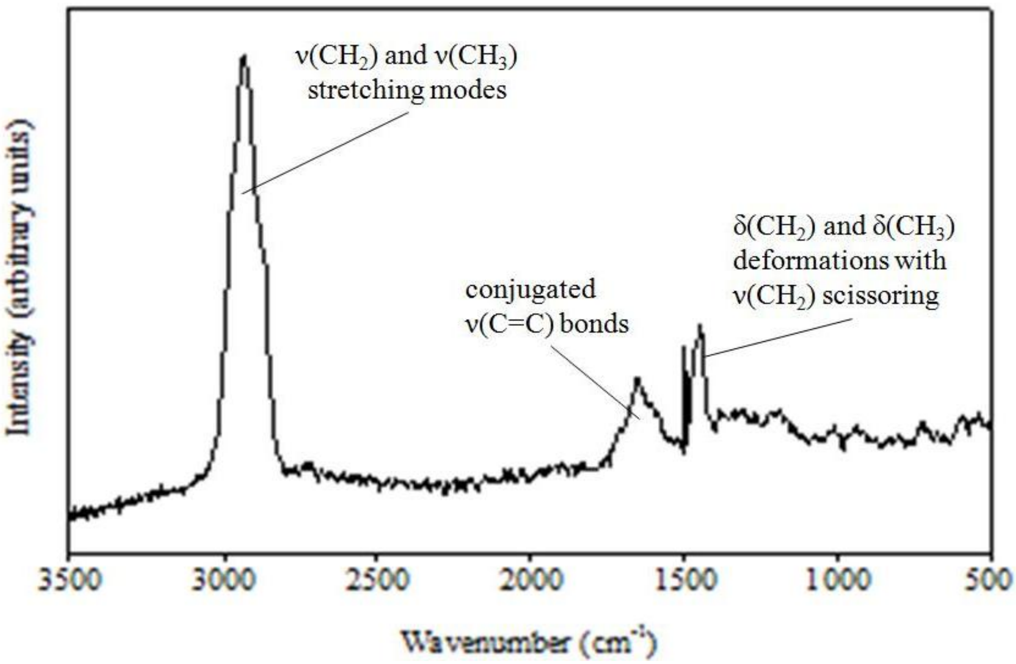
Appendix 6. Tables of results relating to the Case Studies (Chapter 7)

6.1. Case Study 1: Infant, Arrington, Cambridgeshire (7.2)

GC-MS ref	Description	Image	Mass (g)	Results
ARR	Yellow-orange to orange-red fragments. Mostly opaque with some translucency. Brittle and highly friable.		0.6	*Pistacia* spp. resin Key biomarkers: moronic acid oleanonic acid *iso*masticadienonic acid masticadienonic acid + derivatives

Fourier transform Raman spectrum

Wavenumber region 3500-500 cm^{-1}, Nd^{3+}/YAG laser excitation at 1064 nm, nominal laser power 270 mW, 4 cm^{-1} spectral resolution, 1000 scans accumulation.



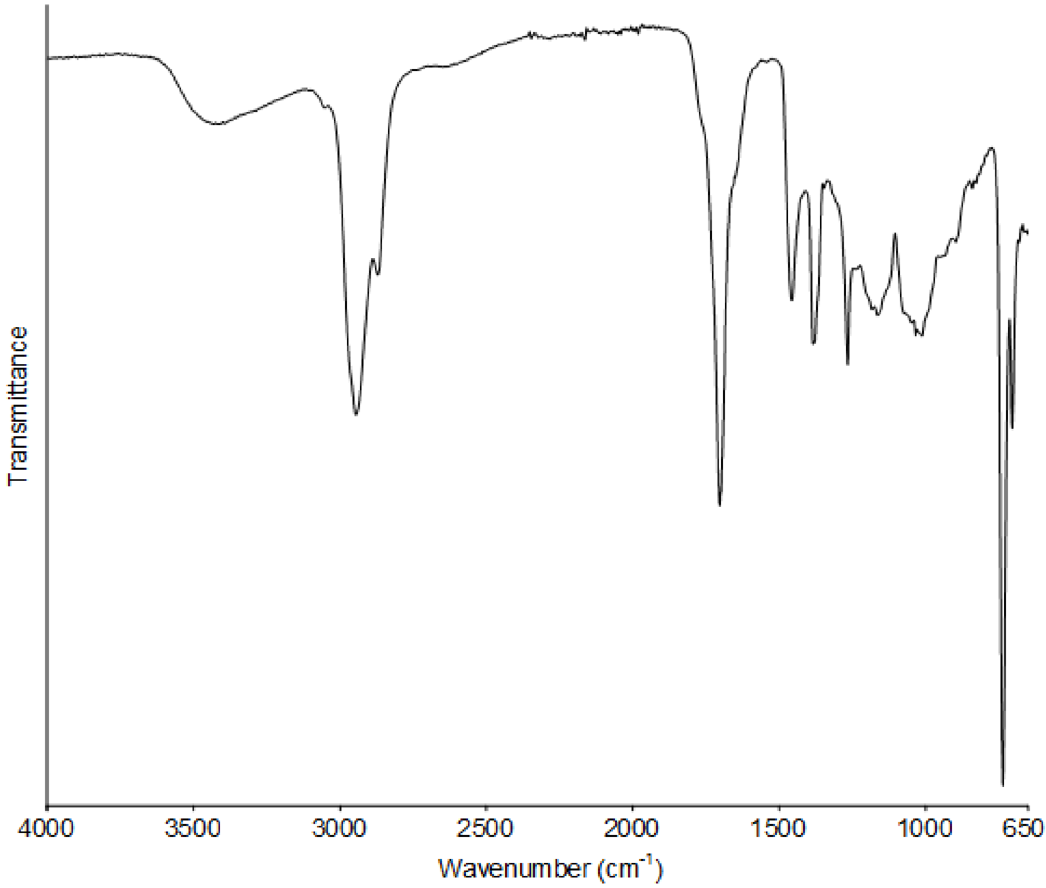
Presence of various hydrocarbon and carbon-carbon bonds:

- Broad strong peak, c. 2930 cm^{-1} : $\nu(CH_2)$ and $\nu(CH_3)$ stretching modes
- Sharp, moderate/weak peak, c. 1440-5 cm^{-1} : $\delta(CH_2)$ and $\delta(CH_3)$ deformations with $\nu(CH_2)$ scissoring
- Medium peak with shoulder, 1649 cm^{-1} : conjugated $\nu(C{=}C)$ bonds characteristic of terpenic acids
- Spike, c. 1495 cm^{-1} : artefact of the instrument

These features are common to most resins and gum-resins.

Attenuated total reflectance-Fourier transform infrared spectroscopy spectrum

Wavenumber 4000-650 cm^{-1}, 16 scans, 4 cm^{-1} spectral resolution



Bands characteristic of polycyclic hydrocarbons with oxygen-containing functional groups:

- a shifted broad stretching (ν) band due to O-H groups ~3500-3300 cm^{-1}
- strong ν(CH_2) and ν(CH_3) bands ~2930-2870 cm^{-1}
- strong ν(C=O) band ~1750-1700 cm^{-1} due to carboxylic acid (COOH) groups*
- ν(C=C) shoulder ~1650 cm^{-1} and other ring vibrations ~1550-1480 cm^{-1};
- CH_2 and CH_3 group bending (δ) ~1460-1450 and ~1375-1385 cm^{-1};
- ν(C-O) bands between ~1275-1240 cm^{-1} and ~1115-1010 cm^{-1};
- C-H out of plane deformations ~880-890 and ~840-820 cm^{-1};
- CH_2 group rocking (ρ) modes ~730-740 cm^{-1}.

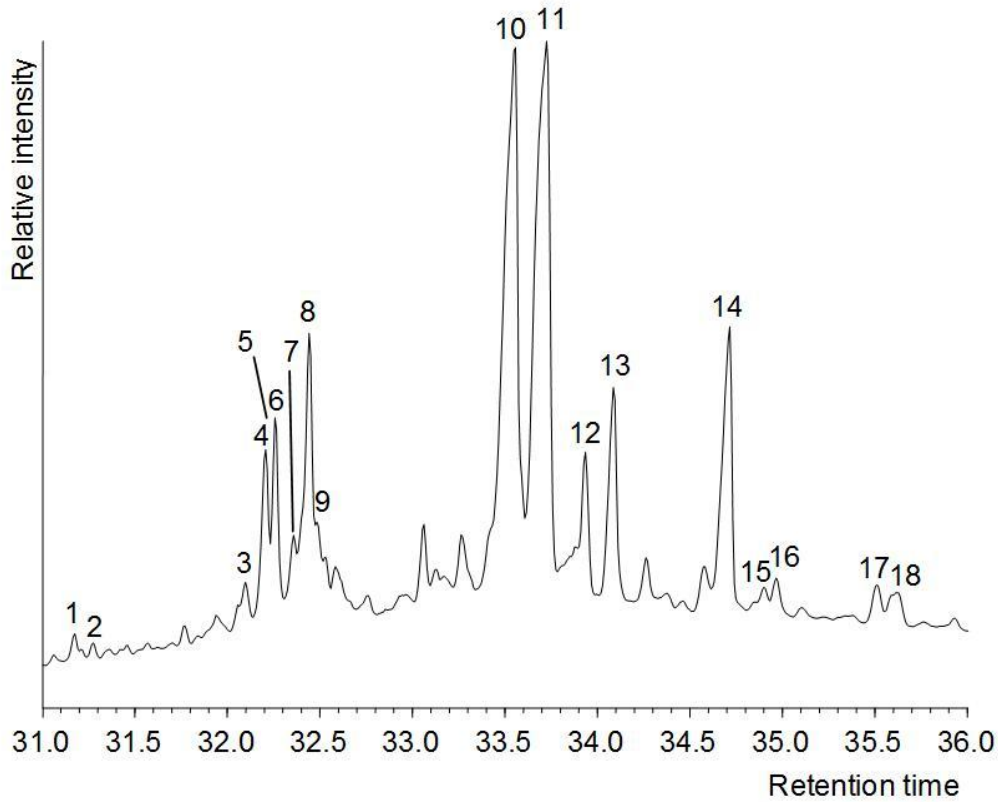
*characteristic of triterpenoid resins

This combination indicates a triterpenoid resin, in particular the C=O stretching band above 1700 cm^{-1}, with the spectrum most similar to that obtained from *Pistacia* spp. resins (Brettell *et al.* 2015c; Bruni and Guglielmi 2014).

Gas chromatography-mass spectrometry

a. methylated

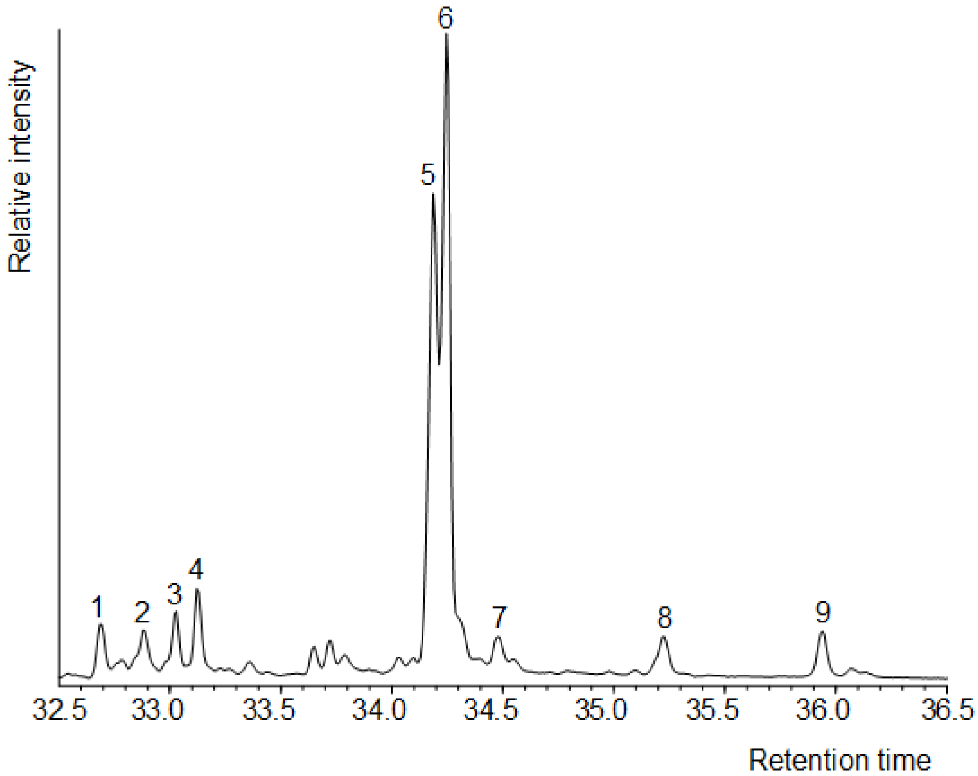
DCM:MeOH (2:1, v/v), methylation (diazomethane, ~0.05 ml, Fales *et al*. 1973). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.



Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
1	442	204	431, 355, 281, 253, 189, 175, 135, 81	3,4-seco-28-norolean-12(18)-en-3-oic acid
2	442	163	427, 405 ,355, 281, 229, 207, 191, 119	3β-hydroxy-6β-hydroxymethyl-28-norolean-17-ene
3	426	411	393, 327, 281, 241, 207, 189, 135, 121	tirucallol
4	410	204	393, 369, 313, 281, 253, 189, 175, 133	28-norolean-12-en-3-one
5	424	218	413, 355, 327, 281, 207, 203, 189, 147	β-amyrenone
6	440	189	413, 281, 262, 251, 207, 203, 163, 133	olean-18-enolic aldehyde (morolic aldehyde)
7	408	408	393, 377, 281, 259, 241, 202, 189, 173	28-norolean-12,17-dien-3-one
8	440	203	413, 262, 251, 218, 189, 175, 133, 119	olean-12-enolic aldehyde (moronic aldehyde)
9	410	163	395, 281, 253, 218, 207, 191, 175, 133	28-norolean-17-en-3-one
10	468	189	453, 409, 391, 262, 249, 203, 163, 119	moronic acid
11	468	203	453, 409, 282, 262, 249, 189, 133, 119	oleanonic acid
12	438	203	409, 257, 232, 189, 175, 133, 119, 105	oleanonic aldehyde
13	468	453	421, 355, 313, 257, 245, 231, 161, 121	*iso*masticadienonic acid
14	468	453	393, 355, 313, 257, 245, 231, 161, 127	masticadienonic acid
15	512	437	497, 355, 341, 315, 241, 301, 189, 121	3α-acetoxy-3-epi*iso*masticadienolic acid
16	470	455	437, 341, 301, 241, 229, 161, 127, 95	3-epi-masticadienolic acid
17	482	217	467, 454, 385, 317, 276, 257, 189, 119	oxidised oleanonic acid derivative
18	512	437	497, 409, 355, 315, 241, 229, 189, 127	3α-acetoxy-3-epimasticadienolic acid

b. silylated

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 μm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.



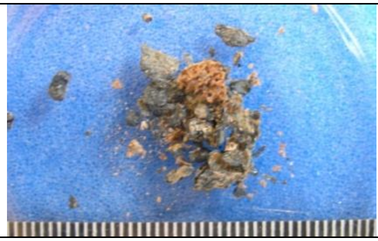
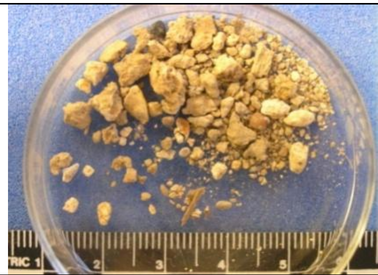
Partial TIC: Arrington sample

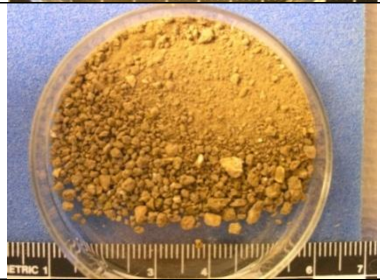
Peak	$M^{+\bullet}$	BP	Key fragment ions	Name of compound
1	410	204	393, 369, 313, 281, 253, 189, 175, 133	28-norolean-12-en-3-one
2	498	218	483, 408, 395, 279, 207, 203>189, 147	β-amyrin
3	410	163	395, 281, 253, 218, 207, 191, 175, 133	28-norolean-17-en-3-one
4	?	73	409, 391, 320, 307, 203, 189, 119	oleanane derivative
5	526	189	511, 409, 391, 320, 307, 219, 203, 133, 119	moronic acid
6	526	203	511, 408, 393, 320, 307, 219, 189, 133, 119	oleanonic acid
7	438	203	409, 320, 232, 189, 175, 133, 119, 105	oleanonic aldehyde
8	526	511	421, 393, 307, 257, 243, 213, 185, 169, 119	*iso*masticadienonic acid
9	526	511	421, 393, 311, 257, 213, 185, 169, 143, 119	masticadienonic acid

6.2 Case Study 2: The cemeteries of Roman London (7.3)

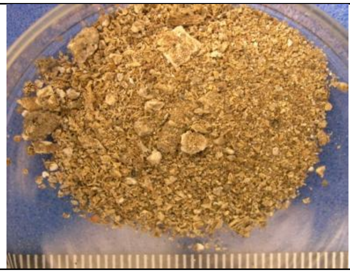
DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

Site/context	GC-MS ref	Description	Image	Mass (g)	Compounds present
Atlantic House, London, EC2 Burial 4 SK216	ATC216L	Dark residue adhering to leg bone fragments		0.3	Plasticisers + other contaminants *n*-alkanes: $C_{19\text{-}33}$ *n*-alkanols: C $_{12\text{-}30}$ (bimodal; C_{16} & C_{26} dom) SFAs: $C_{12\text{-}18}$ (EOP) Steroids: ?coprostanol; cholesterol; β-sitosterol; 5α-cholestanol
	ATC216M	Dark residue in cavity in left side of mandible		0.3	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: C $_{12\text{-}30}$ (bimodal; C_{16} & C_{26} dom) SFAs: $C_{8\text{-}18}$ ($C_{16=18}$) MUFAs: $C_{16:1;\ 18:1}$ Steroids; cholestan-3-one; cholesterol;?β-sitosterol; 5α-cholestanol; stigmastanol
Atlantic House, London, EC2 Burial 7 SK261	ATC261A	Black sticky substance adhering to left acetabulum		0.3	Plasticisers + other contaminants SFAs: $C_{16;18}$ - traces
	ATC261C	Black sticky substance adhering to distal left clavicle		0.3	Plasticisers + other contaminants SFAs: C_{16} - traces

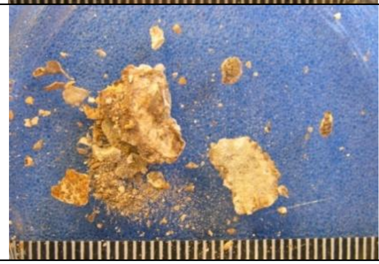
	ATC261F	Black sticky substance adhering to distal right femur		0.3	Plasticisers + other contaminants SFAs: $C_{16;18}$ - traces
St. Martin-in-the-Fields, London	SMFH	Residue from head end of base of stone sarcophagus		2.0	Plasticisers + other contaminants *n*-alkanes: $C_{16\text{-}29}$ *n*-alkanols: $C_{22\text{-}26}$ (C_{24} dom) SFAs: $C_{8\text{-}18}$ (C_{16} dom) MUFAs: $C_{18:1}$ Steroids; cholestan-3-one; cholesterol; cholest-5-en-24-one; β-sitosterol; 5α-cholestanol; campestanol; stigmastanol
	SMFM	Residue from mid-region of stone sarcophagus		1.0	Plasticisers + other contaminants *n*-alkanes: $C_{16\text{-}24}$ - traces SFAs: $C_{16;18}$ (C_{16} dom) Sterols/stanols traces
	SMFF	Residue from foot end of base of stone sarcophagus		0.5	Plasticisers + other contaminants *n*-alkanes: $C_{16\text{-}21}$ SFAs: $C_{8\text{-}18}$ (C_{16} dom) MUFAs: $C_{16:1;\ 18:1}$ Sterols/stanols traces

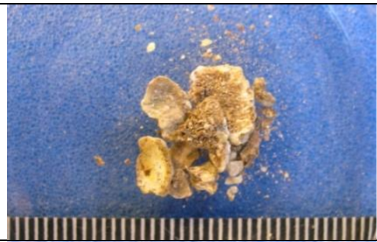
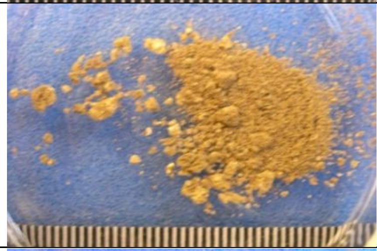
201 Bishopsgate, London, EC2 Burial 377	BGB377P	Yellow-brown residue adhering to pelvic bones		1.0	Plasticisers + other contaminants *n*-alkanes: C_{16-20} - traces *n*-alkanols: none SFAs: C_{12-18} (EOP) - traces
	BGB377S	Yellow-brown masses associated with cranium		2.0	Plasticisers + other contaminants *n*-alkanes: C_{16-20} *n*-alkanols: C_{12-28} (bimodal C_{16} + C_{26}) SFAs: C_{12-18} (EOP) - traces
201 Bishopsgate, London, EC2 Burial 400	BGB400C	Yellow-brown residue adhering to cranial fragments		1.0	Plasticisers + other contaminants *n*-alkanes: C_{15-25} *n*-alkanols: C_{12-28} (C_{18} dom) SFAs: C_{8-18} (C_{16} dom) Branched? traces Steroids: cholesterol; campesterol; 5α-cholestanol
	BGB400L	Yellow-brown residue adhering to leg bones		0.7	Plasticisers + other contaminants *n*-alkanes: traces SFAs: trace C_{16}

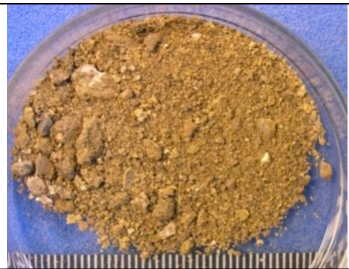
Premier Place, Devonshire Square, Houndsditch, EC2 CDV99, Area 3 Context 146	CDV146a	Yellow-brown silty residue, darker inclusions, right temporal/maxilla		1.6	Plasticisers + other contaminants (major peaks) *n*-alkanes: $C_{15\text{-}28}$ *n*-alkanols: $C_{12\text{-}18;\ 24\text{--}30}$ (bimodal; C_{26} dom) SFAs: $C_{12\text{-}18:0}$ (EOP) Steroids: cholesterol; 5α-cholestanol; ?β-sitosterol
	CDV146b	Fine yellow-brown silty residue, darker inclusions, left distal humerus		0.7	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{12\text{-}18;\ 24\text{--}32}$ (bimodal C_{18}/C_{26}) SFAs: traces Steroids: cholesterol; 5α-cholestanol; ?β-sitosterol; ?stigmastanol
Premier Place, Devonshire Square, Houndsditch, EC2 CDV99, Area 3 Context 147 **CONTROL**	CDV147	Yellow-brown silty residue, dark blocky inclusions, os coxa/ femora		2.0	Plasticisers + other contaminants *n*-alkanes: $C_{15\text{-}30}$ traces *n*-alkanols: $C_{12\text{-}18;\ 24\text{--}30}$ (bimodal C_{18}/C_{26}) SFAs: $C_{8\text{-}18:0}$ (EOP; C_{16} dom) Steroids: cholesterol; 5α-cholestanol; β-sitosterol; stigmastanol
9 St. Clare Street EC1 SCS83 Grave 1 Skeleton 130	SCS130	Brown silt, debris from plastic bags, concretions from hand bones		2.9	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{12\text{-}30}$ (bimodal C_{15}/C_{26}) SFAs: $C_{12\text{-}18:0}$ (EOP; C_{16} dom) Steroids: 5α-cholestane; cholestene x2 isomers; cholesterol; 5α-cholestanol; cholestan-3-one; x3 cholesterol chloride isomers

9 St. Clare Street EC1 SCS83 Grave 2 Skeleton 146	SCS146	Brown silt, debris from plastic bags, post-cranial elements		2.7	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: C_{12-30} (bimodal C_{18}/C_{26}) SFAs: traces
Three Lords Public House, 27 Minories, EC3 TTL85 Grave 7 Skeleton 9	TTL009	Brown sandy silt, debris from plastic bags, skeletal elements		1.0	Plasticisers + other contaminants
Mansell Street, London, E1 Burial 255 SK720	MSL720S	Small areas of dark residues adhering to right scapula and radius		0.06	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: C_{12-18} (C_{12} dom) SFAs: C_{8-18} (C_{16} dom) MUFAs: $C_{18:1}$
East Tenter Street, Scarborough Street, E1 ETN88 Grave cut 39 Context 46	ETN046a	Orange-brown silt with inclusions, compacted within facial bones		3.0	Plasticisers + other contaminants (major peaks) *n*-alkanes: traces *n*-alkanols: C_{12--30} (C_{18} dom) SFAs: $C_{8-18:0}$ (C_{16} dom); MUFAs: $C_{18:1}$

	ETN046b	Orange-brown silt, debris from plastic bags, post-cranial elements		1.5	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: C_{12--30} (bimodal C_{18}/C_{26}) SFAs: $C_{12-18:0}$ (C_{14} dom)
Armagh Road, E3 AR72 Context 1	AR72/1a	Pale-brown silt, grey-brown aggregations, associated with cranium		4.0	Plasticisers + other contaminants *n*-alkanols: C_{12-30} traces + SFAs: traces Steroids: cholesterol; 5α-cholestanol Deg. bone: diploptene; diplopterol Bitumen: extensive homologous series of *n*-alkanes (OEP); pristane + phytane; steranes C_{27-29} (3-4 isomers of each); hopanes C_{27-30} (C_{30} dom)
	AR72/1b	Pale-brown silt, grey-brown aggregations, post-cranial elements		3.0	Plasticisers + other contaminants *n*-alkanols: C_{12-32} (bimodal; C_{12} + $_{26}$ dom) SFAs: $C_{12-18:0}$ (EOP; C_{16} dom) Steroids: cholesterol; 5α-cholestanol Deg. bone: diploptene; diplopterol Bitumen: *n*-alkanes C_{17-33} (OEP); pristane + phytane; steranes traces; tricyclic polyprenanes C_{23-25}; hopanes C_{27-30} (C_{30} dom) ?Resin: traces of moronic acid; oleanonic acid; 28-nor-olean-17-en-3-one – could be from bitumen

Armagh Road, E3 AR72 Context 3	AR72/3	White-brown mixture of silt + plaster, post-cranial skeletal elements		4.0	Plasticisers + other contaminants *n*-alkanes: C_{17-23} (OEP C_{19} dom) *n*-alkanols: C_{12-32} (C_{18} dom) SFAs: traces Steroids: cholesterol; 5α-cholestanol
Great Dover Street, London Burial 26 SK150	GDV150R	White residue adhering to ribs		0.3	Plasticisers + other contaminants
	GDV150V	White residue adhering to vertebrae		0.3	Plasticisers + other contaminants
Great Dover Street, London Burial 17 SK126	GDV126T	White residue adhering to ribs and cervical vertebrae		0.2	Plasticisers + other contaminants
Great Dover Street, London Burial 25 SK178	GDV178C	White residue adhering to inner surface of cranial bones		0.2	Plasticisers + other contaminants *n*-alkanes: traces SFAs: $C_{16;\ 18}$ - traces Cholesterol derivatives

	GDV178F	White residue adhering to foot bones		0.2	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{16\text{-}26}$ SFAs: $C_{10\text{-}20}$ (EOP); MUFAs: $C_{16:1;\ 18:1}$ Steroids: cholesterol; campesterol; 5α-cholestanol; cholesterol derivatives
Great Dover Street, London Burial 27 SK242	GDV242C	Yellow residue adhering to shaft of left clavicle		0.2	Plasticisers + other contaminants
	GDV242L	Yellow residue adhering to left tibia and fibula		0.5	Plasticisers + other contaminants *n*-alkanes: $C_{14\text{-}30}$ *n*-alkanols: $C_{12\text{-}18}$ SFAs: $C_{8\text{-}18}$ (EOP; C_{16} dom.) MUFAs: $C_{16:1;\ 18:1}$ – traces Cholesterol derivatives
	GDV242P	White residue adhering to inner right parietal		0.1	Plasticisers + other contaminants SFAs: $C_{12\text{-}18}$ (EOP; C_{16} dom.) Cholesterol derivatives

Lant Street LTU03 Context 292	LTU292	Very fine orange-brown silty/sandy residue, debris from plastic bags + post-cranial skeletal elements		3.0	Plasticisers + other contaminants *n*-alkanols: $C_{12\text{-}18;\ 24\text{--}30}$ (bimodal C_{18}/C_{26}) SFAs: $C_{12\text{-}18:0}$ (EOP C_{16} dom) MUFAs: $C_{18:1}$ Steroids: cholesterol; 5α-cholestanol; cholesta-3,5-dien-7-one
Lant Street LTU03 Context 334	LTU334	Orange-brown silty/sandy residue, debris from plastic bags + post-cranial skeletal elements		2.7	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{12\text{-}18;\ 24\text{--}30}$ (bimodal C_{16}/C_{26}) SFAs: $C_{12\text{-}18:0}$ (EOP; C_{16} dom) Steroids: cholesterol; 5α-cholestanol; cholesa-3,5-dien-7-one
Lant Street LTU03 Context 385	LTU385	Orange-brown sandy residue, debris from plastic bags + post-cranial skeletal elements		3.0	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{12\text{--}30}$ (bimodal C_{16}/C_{26}) SFAs: $C_{14\text{-}18:0}$ (EOP; C_{16} dom) Steroids: cholesterol; 5α-cholestanol; cholesa-3,5-dien-7-one
Lant Street LTU03 Context 434	LTU434	Very fine orange-brown sandy residue, compacted within cranium		3.0	Plasticisers + other contaminants *n*-alkanols: traces SFAs: $C_{8\text{-}18:0}$ (EOP; C_{16} dom)

Appendix 6.2.1 Cemeteries to the west of London

6.2.1.1 Overview

The western cemetery was focussed around modern Holborn and Smithfield, close to the city wall between Newgate and Aldersgate. Although many of the finds recovered by antiquarians were not curated, forty-three sites have been identified and indicate that the area was in use from the 1st to early 5th centuries AD. In total more than 171 inhumation burials have been recorded in this western cemetery group (Watson 2003: 7-9). They include four interments within stone sarcophagi, one of which was reported to be a plaster burial while another contained a double inhumation (male and female placed head to toe). Near the latter, the skeletal remains of a female were found in a stone sarcophagus with inner lead coffin. At least two other lead coffins were also recovered, one of which was decorated and contained two juveniles (Hall 1996). Unfortunately, none of these appear to be extant although a more recently excavated sarcophagus from St. Martin-in-the-Fields (City of Westminster, WC2) and normative burials where unusual residues had been reported from the site of Atlantic House (Holborn, EC1) were accessible.

6.2.1.2 Site details and results: St. Martin-in-the-Fields

Three samples were selected from the base of a large limestone sarcophagus discovered during work at St. Martin-in-the-Fields, City of Westminster, WC2 (**Figure 6.2.1**). This famous church is situated at a considerable distance from the western boundary of *Londinium* on the site of a burial ground in use during the Roman and medieval periods. Many of the finds from this area were removed during the 19th century AD but more than twenty Roman period inhumations, including the sarcophagus, have recently come to light. Dated to the late 4th century AD, this container had been damaged during the building of a Victorian sewer and the body disturbed (Colman Getty 2006). The human remains and associated environmental (grave deposit) samples were not able to be accessed (awaiting accessioning to the MoL). Moreover, the latter were thought to have already been processed using industrial methylated spirits thereby removing most, if not all, organic traces of archaeological relevance. The samples from the base of the sarcophagus, which had previously been on display, contained a range of ubiquitous lipid moieties: *n*-alkanes, *n*-alkanols; SFAs, MUFAs, sterols (cholesterol and derivatives; *β*-sitosterol) and stanols (5α-cholestanol; campestanol; stigmastanol).

Figure 6.2.1. Sarcophagus, St. Martin-in-the-Fields, City of Westminster, London, UK (Author).

6.2.1.3 Site details and results: Atlantic House

Five samples were collected from two burials from the site at Atlantic House, Holborn Viaduct (ATC97), London, EC1. Located beside the River Fleet along a major road leading to the west, linking London with Silchester, this area seems to have been used as a burial ground between the mid-2nd and early 5th centuries AD. A total of twenty-nine cremation and nineteen inhumation burials were recovered, many or which had been disturbed, although most seemed to have been interred in wooden coffins (Watson 2003: 15-18). Dark staining was observed around the skeleton of a partially flexed, near edentulous, elderly male denoted Burial 4 (Context 216) who was accompanied by a Black Burnished ware ceramic vessel dated c. 140-160 AD and the remains of a chicken. Black tar-like concretions were also noted on the skeletal remains of Burial 7 (Context 261), a young adult male whose grave fill contained eighty-one ceramic sherds dated c. 140-250 AD and a cattle horn core (Watson 2003: 40-41).

These two burials were sampled to address the hypothesis that resinous substances were more widely utilised and not just reserved for those accorded more elaborate rites. It should be noted, however, that due to the relatively unprotected nature of such inhumations absence of evidence does not necessarily constitute evidence of absence. As no samples of the stained soil associated with Context 216 could be located, residues were selected from poorly preserved leg bone fragments (ATC216L) and from within the left mandibular foramen (ATC216M; **Figure 6.2.2a**). These contained low levels of *n*-alkanes, *n*-alkanols, saturated (SFAs) and monounsaturated (MUFAs) carboxylic acids, sterols (cholesterol; *β*-sitosterol) and stanols (5α-cholestanol; stigmastanol).

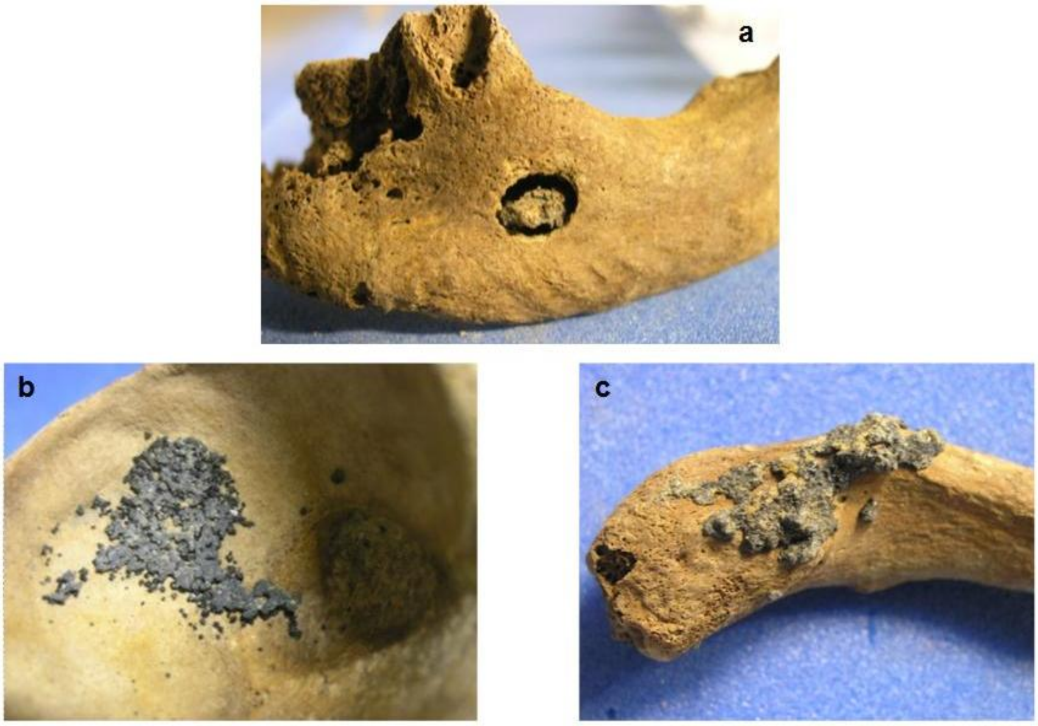


Figure 6.2.2. Residues from inhumation burials, Atlantic House, London: a. within enlarged mandibular foramen, Burial 4; b. adhering to acetabulum, Burial 7; c. on clavicle, Burial 7 (Author).

Three samples of a black tar-like substance distributed in patches across the skeletal remains of Burial 7 were collected for analysis. These consisted of residues adhering to the distal right femur (ATC261F), left acetabulum (ATC261A; **Figure 6.2.2b**) and lateral left clavicle (ATC261C; **Figure 6.2.2c**). With the exception of traces of the most ubiquitous SFAs ($C_{16:0}$ and $C_{18:0}$) and a range of phthalate plasticisers, no extractable lipids were present. Evaluation of polymerised components is recommended.

Appendix 6.2.2 The northern cemetery

6.2.2.1 Overview

Ermine Street, the main road to the north which left *Londinium* through Bishopsgate was flanked by burials that spanned the whole of the Roman period and also extended westwards into the region of Moorgate and Finsbury Circus (Barber and Bowsher 2000: 3). Discoveries from this cemetery date back to 1576 with many graves excavated prior to 1900 during the construction and development of Liverpool Street Station and the surrounding areas. Only those that contained significant grave goods or were interred in substantial containers (coffins or urns) were generally recorded so information regarding the vast majority has been lost (Hall 1996). In this burial ground, as elsewhere in south-east England, it seems that the predominant rite during the 1^{st}-2^{nd} centuries AD was cremation with some individuals continuing to choose this method of disposal into the 3rd-4th centuries AD. Inhumation burials, dated as early as AD 100, have also been recovered with this mortuary practice having steadily increased in popularity and continued until the end of the Roman period (Hall 1996).

Antiquarian reports record at least eighty-seven inhumations including one in a decorated lead coffin and four in stone sarcophagi (one of which contained the remains of two individuals). Three of the latter may have been plaster burials (two from Stothard Place, one from Cutler Street) while two had been placed under an arched vault and so appear to bear some similarity to the recent find from Naintré, near Poitiers (Devièse *et al.* 2011; **3.3.4**). Little remains of these finds although, from surviving collections, it appears that the northern cemetery had the highest number and widest variety of ceramic types in comparison with the other burial grounds of London. Likewise, a considerable variety of high quality grave goods are extant although there is rarely any record regarding the related burial (Hall 1996). Needless to say, organic materials associated with these burials do not survive.

Recently, however, a number of controlled excavations have been undertaken. These include extensive work in the area of Bishopsgate which has uncovered around 100 Roman period burials, many of which were accompanied by a range of grave goods (Swift 2000; Thomas *et al.* 2003). The earliest interments in this area consisted of inhumations mostly on a north-south orientation with later phases characterised by individuals predominantly aligned in rows with head to the west. A number of these have been identified as chalk burials (Swift 2000) while in Area 10 of the 280 Bishopsgate and Spitalfields Ramp excavations the final row to the south contained five more substantial burials placed on a slight rise. Three of these were represented by broken sarcophagi which had been robbed in antiquity. Another was found to contain a child encased in plaster within a timber-lined mausoleum accompanied by eight glass vessels and the last (detailed in **7.4**) was a complete stone sarcophagus containing an undamaged lead coffin (Thomas 1999; Thomas *et al.* 2003).

6.2.2.2 Site details and results: 201 Bishopsgate

Four samples were collected from two burials found at 201 Bishopsgate (BGB98), London, EC2. This site lay beside Ermine Street to the east of the Walbrook Stream with six burials recovered from the area excavated (Swift 2000: 1-2). The two graves sampled were those of a mature adult male (B377) with a healed rib fracture and age-related degenerative changes and that of a child (B400) of about 7-8 years old which was dated to the late 3rd-4th centuries AD and cut B377. Both individuals had been buried supine, extended in wooden coffins on beds of crushed chalk and were orientated north-south. The child was accompanied by three copper alloy bracelets and some fine wire near the right ankle (Swift 2000: 18-19).

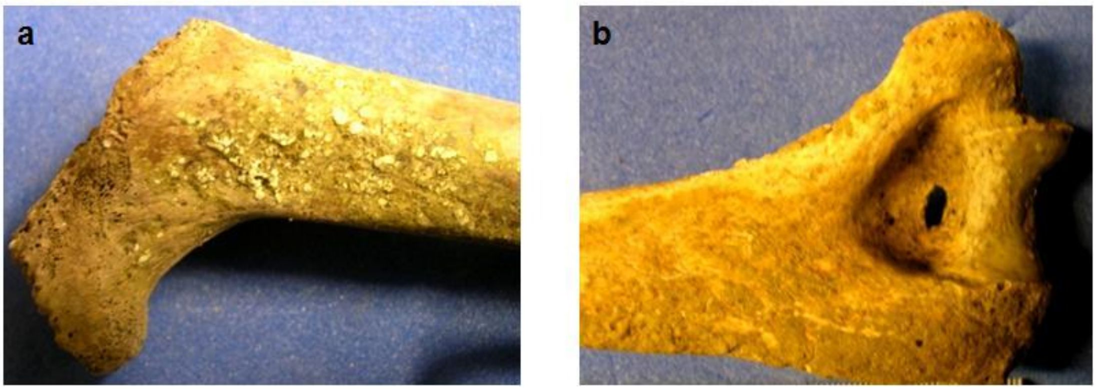


Figure 6.2.3. Residues sampled: a. left proximal femur, Burial 400, 201 Bishopsgate, London; b. left distal humerus, CDV146, Premier Place, Houndsditch, London (Author).

Two samples of the yellow-brown concretions on both sets of remains were selected for analysis, one from the lower body and one from the cranial area of each individual (**Figure 6.2.3a**). Trace levels of ubiquitous *n*-alkanes (C_{16-20}), *n*-alkanols (C_{12-28}) and SFAs ($C_{8:0-18:0}$) were present in the samples from B377. Likewise, the residue from the cranium of B400 contained a range of these common components together with an input from degraded animal and/or faecal matter denoted by the presence of cholesterol, campesterol and 5α-cholestanol (cf. Bull *et al.* 2002; Evershed *et al.* 1997b). The sample from the leg bones of B400 was almost devoid of lipids.

6.2.2.3 Site details and results: Premier Place

In 1999, part of a Roman burial ground was discovered at Premier Place (CDV99) at the junction of Cutler Street, Houndsditch and Devonshire Square, EC2. Thirty-five graves containing thirty-six inhumations (an adult female and a neonate were in a single grave) were recovered. The majority appeared to have been interred extended, supine in wooden coffins and orientated west-east (with two exceptions). The graves were arranged in rows and respected one another suggesting that they were marked above ground in some way. As they were aligned parallel to the city boundary, they probably post-date AD 120 (Sankey and Connell 2008). The regular arrangement, absence of cremations and fact that a number of the burials contained plaster is more in character with late Roman mortuary practices (Barber and Bowsher 2000; 104, 116-7; Philpott 1991: 90-96). However, as none of these burials contained grave goods this cannot be confirmed. All age groups and both sexes were represented (Sankey and Connell 2008).

Having ascertained that no environmental samples had been collected and that many of the remains were severely truncated, fragmentary and poorly preserved, two burials were selected for assessment. Thus, two samples were collected from Context 146, one of the more complete skeletons recovered (**Figure 6.2.3b**). This mature adult female who had suffered a Colles fracture of the distal radius and broken a right 1st metatarsal, probably as the result of a fall, had been encased in or laid on a bed of plaster within a wooden coffin. A number of ceramic sherds were found in the grave fill and one in the mouth (residual). A sample was also obtained from Context 147 as a control since this mature adult female had been interred without plaster and possibly without a container of any kind (Sankey and Connell 2008).

All three samples were contaminated by phthalate plasticisers, probably derived from the plastic bags in which they had been stored. Those from Context 146 contained little organic matter with the lipid species identified from both burials predominantly end-products of widespread occurrence, for example, *n*-alkanes, *n*-alkanols and SFAs ($C_{12:0\text{-}18:0}$). The sterols (cholesterol; *β*-sitosterol) and stanols (5α-cholestanol; stigmastanol) present indicate an input from higher plant and mammalian sources.

Appendix 6.2.3 The north-east and eastern cemeteries

6.2.3.1 Overview

Treated as one continuous cemetery by Hall (1996), subsequent research has suggested that the region to the east of the walls of London contained at least two separate cemetery areas, one aligned along the main road from Aldgate to Colchester and a second that spread either side of a road which may have stretched from the Minories to Wapping Lane or even as far as (Brod)Love Lane, E1 (Barber and Bowsher 2000: 51-52, 58). A number of antiquarian reports and modern excavations record Roman period burials from these eastern cemeteries which now lie in the London Borough of Tower Hamlets (Barber *et al.* 1990; Barber and Bowsher 2000; Ellis 1985; Evans and Pierpoint 1986; Whytehead 1986; **Figure 6.2.4**). Their use seems to extend from the late 1st century AD to the end of the Roman occupation with around 165 cremations and 700 inhumations recovered to date (Barber and Bowsher 2000: 58, 82, 229-300; Hall 1996).

The majority of the inhumation burials were extended, supine in wooden coffins within rectangular grave cuts. Two major alignments, north-south and east-west, with head to either end, were observed within a chronological trend towards an increase in east-west aligned inhumations (Barber and Bowsher 2000: 82-85). In comparison with other Roman cemetery populations, only a small percentage of those interred to the east of London seem to have been buried with grave goods (Barber and Bowsher 2000: 117-141; Evans and Pierpoint 1986; Hall 1996). Osteological analysis indicates a community in which many were involved in manual labour but who had an adequate level of nutrition and hygiene and displayed relatively low levels of interpersonal violence (Barber and Bowsher 2000: 285-287).

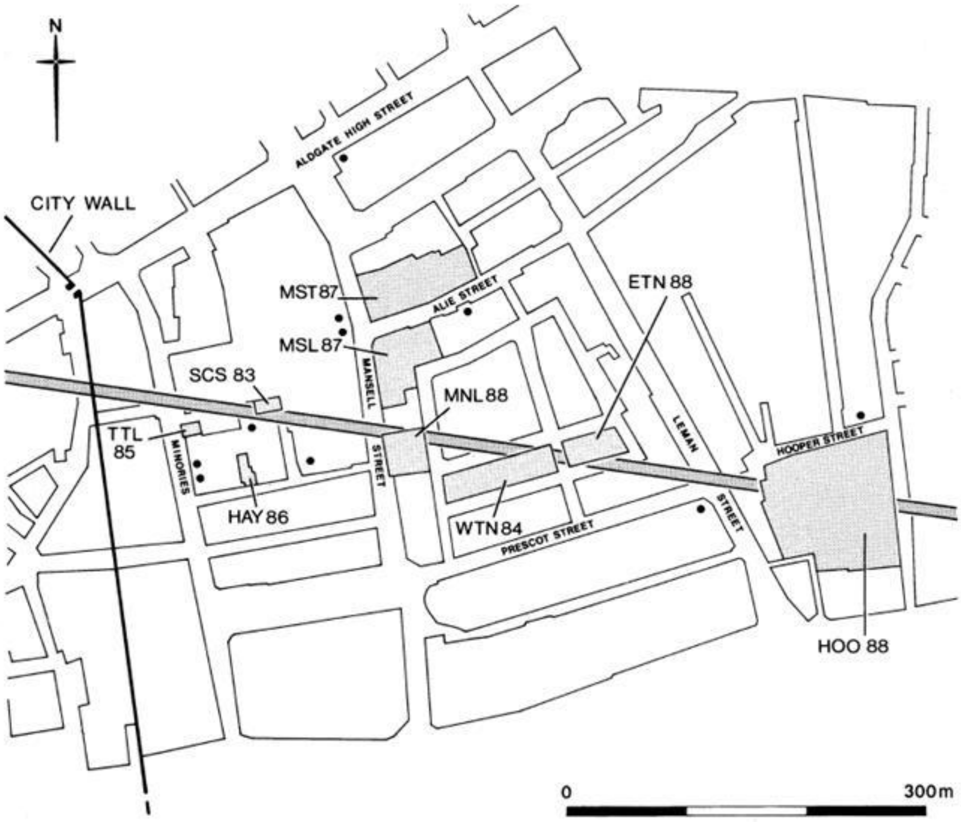


Figure 6.2.4 Location of excavations, eastern cemetery, London (Figure from Barber *et al.* 1990: Figure 2, 3).

No stone sarcophagi and only two lead coffin burials have been recovered in recent excavations, although a further four lead and three stone containers are described in antiquarian reports (Barber and Bowsher 2000: 95-96, 336-338; Hall 1996). A significant number of burials (c. 90) containing a white substance have also been recorded. This material encased the body of some individuals while others had seemingly been laid on a bed of the substance and a few had small patches in various positions on the body. These plaster burials, as discussed by Barber and Bowsher (2000: 101-104), appear to extend throughout the life of the cemetery with some slight increase in the later periods. They show no particular correlation with body orientation or biological sex although they seem more common in the burial of infants and young adults. Analysis of samples from five burials using X-ray diffraction (XRD) and scanning electron microscopy (SEM) showed that this substance was calcium carbonate which contained coccoliths typical of marine sedimentary chalk. As calcining or slaking would probably have destroyed such evidence (dependent on the temperature achieved), the chalk may have been mixed with water and applied as slurry. Previous examination of white materials from similar burials around Roman London concluded, however, that slaked lime (calcium hydroxide) derived from marine limestone, possibly chalk, had been used (Whytehead 1986).

6.2.3.2 Site details and results: eastern burial ground

Two burials in decorated lead coffins probably placed within wooden containers were discovered during excavations at 49-59 Mansell Street, London, E1 (MSL87, Barber and Bowsher 2000: 95). Unfortunately the remains of the child (c. 6-12 years old) from Burial 392 (SK754; F361) interred with a range of high quality grave goods dated c. AD 250-350 including glass artefacts, an ivory figurine, three pipeclay Venus figurines, a pair of gold earrings and a bone pyxis (small circular container with lid) could not be located. A single sample was, however, collected from Burial 355 (SK720; F351), the inhumation of a robust young adult male (c. 16-25 years old), dated between AD 180-400, who had been laid on a bed of 'chalk'. This sample consisted of traces of a dark residue adhering to the right scapula and radius. The only lipids identifiable, aside from phthalates, were traces of *n*-alkanes, *n*-alkanols (C_{12-18}), SFAs ($C_{12:0-18:0}$, evens) and the MUFA $C_{18:1}$.

At East Tenter Street (ETN 88), eight inhumations, three of which were interred with plaster, were discovered in 1988 alongside evidence of a mortared flint structure, possibly a mausoleum, and grave goods indicative of a 3rd century AD date (Barber and Bowsher 2000: 29). From 9 St. Clare Street (SCS 83), four inhumations and one cremation burial were recovered in 1983 from the precinct of the abbey of St. Clare. These were dated between the 1st-3rd/4th centuries AD on the basis of associated finds and their relative stratigraphy. One, Grave 4, had a ragstone surround while the foundations of a possible mortuary structure were observed nearby (Ellis

1985). In addition, again from within the boundaries of the abbey, a single plaster burial in a wooden coffin was found at the Three Lords Public House site, 27 Minories, EC3 (TTL85). Aligned NS (head to the north), pottery from within the fill indicated a 3rd-4th century date although the remains themselves were severely truncated (Museum of London, unpublished site record).

As no environmental samples were extant, portions of the loose debris associated with three inhumations: the individual from the Three Lords site (TTL009), Context 130, 9 St. Clare Street (SCS130), and Skeleton 46, East Tenter Street (ETN046b) were collected for analysis together with compacted material from the cranium of the latter (ETN046a). A sample from Context 146, 9 St. Clare Street (SCS146) was chosen to provide a comparative control. Insufficient material was present in the other two plaster burials from East Tenter Street. Again, these samples were found to be dominated by phthalates, with some traces of *n*-alkanes, *n*-alkanols (C_{12-30}), SFAs ($C_{12:0-18:0}$, evens) and MUFAs ($C_{18:1}$) and cholesterol derivatives (in SCS130).

*Site details and results: Armagh Road, Bow, Tower Hamlets (see **7.3.3.2**)*

Appendix 6.2.4 The southern cemeteries

6.2.4.1 Overview

The southern cemeteries of Roman London were located in the region of Southwark. Activity in this area developed along the waterfront with timber workshops established in the 1st century AD. During the 2nd century AD, it seems to have become a mixed residential and commercial suburb with the erection of public buildings including a bath-house and more substantial warehouses. A villa complex may have been added in the 3rd-4th century AD (Cowan 2003: 12-70). The burials around Southwark appear to have been focused in two distinct areas. The first group were situated to the south of the junction of Stane Street and Watling Street and along the sides of these roads while the second group may have been aligned along a road thought to have run from Southwark Bridge to Lambeth. Although many mausolea seem have been present, there are no records of any stone sarcophagi from these cemeteries and only one decorated lead coffin whose current whereabouts are unknown (Hall 1996). As in the eastern cemetery, a number of individuals interred in wooden coffins were either laid on a ‘bed’ of white material or encased in a white substance (Cowan 2003; Dean 1981; Dean and Hammerson 1980; Mackinder 2000).

6.2.4.2 Site details and results: Courage Brewery

Seven inhumation burials were recovered from the Courage Brewery site (COSE84), Park Street, SE1 which was used as a burial ground in the late 4th century AD. These individuals had been interred in wooden coffins. Three of the graves reportedly contained plaster (Cowan 2003: 70-73; 191-194). Soil samples collected during excavation could not be located and examination of the skeletal remains showed that no adhering residues remained.

6.2.4.3 Site details and results: Great Dover Street

The site at 165 Great Dover Street (GDV96). London, SE1 is situated beside Watling Street, approximately 1 km to the south of the River Thames, outside the core area of the Roman suburb of Southwark (Mackinder 2000: 1-2). The earliest activity in this area dates to the 1st century AD when it may have been part of a farm. A number of small enclosed and unenclosed roadside cemeteries containing both cremation and inhumation burials were then established from the mid-2nd century onwards. Ornate tombs and monuments appear to have been present as a range of carved stone items, including a large pinecone finial, were discovered together with a small temple-like building (Mackinder 2000: 5-19).

Twenty-five inhumations dating from the 2nd-3rd centuries AD were recovered from these burial grounds. Many of these individuals were sub-adults and young adults and most had been interred supine, extended, in wooden coffins although two were prone and one had been decapitated. In addition, three graves (Burials 25-27) had larger than average grave cuts with sides that may have been supported by wooden shuttering. These contained inhumations in wooden coffins packed with a white material while a fourth individual (Burial 17) had been laid on a bed of a white substance (Mackinder 2000: 19-21).

Eight samples were collected from these four burials to address the hypothesis that resinous substances may be correlated with the presence of plaster even in the absence of more substantial containers. The samples obtained from Burial 17, Burial 26 and from the left clavicle of Burial 27 were essentially inorganic and contained only phthalate plasticisers and related contaminants. The remaining four samples, from the lower leg bones and right parietal of Burial 27 and both samples from Burial 25, provided various traces of *n*-alkanes, *n*-alkanols, SFAs ($C_{16:0}$; $C_{18:0}$) and MUFAs ($C_{16:1}$; $C_{18:1}$). In addition, the residue from the foot bones of Burial 25 contained traces of cholesterol, campesterol and 5α-cholestanol and a series of unidentified cholesterol derivatives (c. 29-32 min).

6.2.4.4 Site details and results: Lant Street

Excavations at 52-56 Lant Street (LTU03), Southwark, SE1 in 2003-2004 revealed the presence of two cremation and eighty-four inhumation burials. Located to the west of Stane Street, two main phases of use were identified within the area investigated. In the northern part of the site the earliest burial appears to have been that of a neonate interred with a complete 1st century ceramic jar and somewhat separated from the remaining eleven individuals. The latter date from the 2nd-early 3rd century AD and may have been situated on the periphery of a more extensive cemetery to the north. They included two prone burials and one unusual triple inhumation of a young adult male, 9 month old infant and 4-5 year old child. Another infant and two dog burials, one apparently wearing a collar, were also discovered (Ridgeway *et al.* 2013: 13-14). The second burial phase in the central and southern area of the site again appears to have been part of a larger cemetery and has been dated to the late 3rd-early 5th century AD. The majority, here, had been interred supine, extended in wooden coffins with a range of grave goods, some of considerable quality. A number of individuals had been interred with varying amounts of plaster (Ridgeway *et al.* 2013: 16-23).

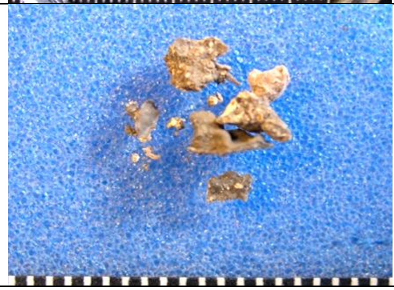
Samples were collected from four of the plaster burials. Although these appeared to be sandy soil which would be inimical to the survival of organic matter, assessment was considered important due to the exotic grave goods found with BL15 and potential for this individual and BL33, based on their oxygen isotope ratios, to have been migrants (Ridgeway *et al.* 2013: 113-114). Thus, the burials assessed comprised:

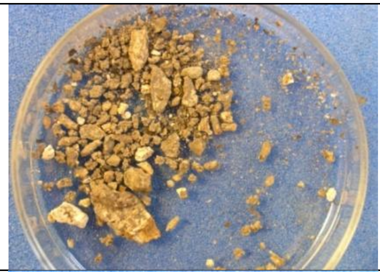
- BL15 (LTU385), an adolescent (c. 14 years old) who had suffered bouts of illness during childhood as reflected in severe enamel hypoplasia. These abnormalities included heavy pitting, 'sleeve-like' deficiencies and irregularities of the molars together with extensive caries. A supernumerary talon cusp (1^{st} left incisor), possible indications of rickets affecting the arm bones and some unusual morphologies in the tarsals were also observed. This individual had been interred supine, extended with some exceptional, imported grave goods including two glass vessels dated to the late 2^{nd}-3^{rd} centuries AD, bone inlay plaques and copper-alloy fittings indicative of a decorative casket, a copper-alloy key and a, on a copper-alloy chain, a folding knife with an ivory handle in the form of a leopard (Ridgeway *et al.* 2013: 37, 44-46, 113-114);
- BL33 (LTU434), an adult female who had suffered a fracture of the left olecranon which led to infection (denoted by the formation of a cloaca) and osteoarthritis of the left elbow. Degenerative changes to two thoracic vertebrae and ante-mortem loss of four teeth had also affected this individual who had been deposited supine, extended within a wooden coffin accompanied by four ceramic vessels and a coin indicative of burial in the early 4^{th} century AD (Ridgeway *et al.* 2013: 83);
- BL39 (LTU334), an adult female interred supine, extended in a wooden coffin and accompanied by three 3^{rd}-4^{th} century AD decorated Nene Valley colour coated ware ceramic beakers and a copper-alloy plate fragment (Ridgeway *et al.* 2013: 85);
- BL43 (LTU292), a middle adult female placed supine, extended in a wooden coffin alongside a copper-alloy bracelet with a crenellated edge and a hairpin (Ridgeway *et al.* 2013: 86).

Again, these samples contained only phthalate contaminants and ubiquitous (*n*-alkanes, *n*-alkanols, SFAs), low abundance end products of the degradation of plant and/or animal tissues.

6.3 Case Study 3: The 'Spitalfields Lady', London, E1 (7.4)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

Site/context	GC-MS ref	Description	Image	Mass (g)	Compounds present
SRP98 Context – SK15903 Materials associated with skeleton	SPF OR	Fine yellow-brown layer adhering to a number of skeletal elements		0.05	Plasticisers + other contaminants *n*-alkanes: C_{18}-C_{22} *n*-alkanols: C_{14}-C_{18}
	SPF BD1	Sandy-gravel mixture from bags containing the human remains		2.0	Plasticisers + other contaminants *n*-alkanes: C_{17}-C_{26} *n*-alkanols: C_{10}-C_{18} SFAs: $C_{12\text{-}16}$ traces
	SPF HY	Dark brown mass adhering to inner surface of hyoid bone		0.03	SFAs: C_8-C_{16}, C_{18} (C_{16} dom.>C_{12}>$_{14}$>$_{18}$) Triterpenic compounds – possibly *Pistacia* spp.: β-amyrin; 28-norolean-17-en-3-one; moronic acid; oleanonic acid; oleanonic aldehyde

SRP98 Context 15900 – between lead coffin and stone sarcophagus Flotated in IMS	F289	Gravel, sand, vegetation		0.4	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: traces SFAs: $C_{16; 18}$ – traces Cholesterol - trace
	F290	Gravel, sand, vegetation Strong purple coloration to solvent extract		0.3	Plasticisers + other contaminants *n*-alkanols: $C_{14\text{-}18}$ (EOP) - traces SFAs: $C_{12\text{-}18}$ – traces
	F291	Gravel, sand, vegetation		0.9	Plasticisers + other contaminants
	F292a	Gravel, sand, vegetation		1.0	Plasticisers + other contaminants *n*-alkanes: $C_{15\text{-}33}$ *n*-alkanols: $C_{12\text{-}30}$ (bimodal C_{16}/C_{26}) SFAs: $C_{12\text{-}18}$ (EOP; C_{16} dom) Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol Triterpenoids: frags.189/203; Mol. ion 526; ?*Pistacia* spp.

	F292b	Gravel, sand, vegetation Blue/black coloration to solvent extract when concentrated		1.0	Plasticisers + other contaminants *n*-alkanes: $C_{15\text{-}31}$ *n*-alkanols: $C_{12\text{-}30}$ (bimodal C_{16}/C_{26}) SFAs: $C_{12\text{-}18}$ (EOP; C_{16} dom) MUFAs: $C_{16:1;\ 18:1}$ Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol
	F293	Gravel, sand, vegetation Blue/black coloration to solvent extract when concentrated		1.0	Plasticisers + other contaminants *n*-alkanes: $C_{15\text{-}27}$ *n*-alkanols: $C_{12\text{-}30}$ (bimodal C_{16}/C_{26}) SFAs: $C_{16;\ 18}$ (C_{16} dom) MUFAs: $C_{18:1}$ Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol
	F294	Gravel, sand, vegetation Blue/black coloration to solvent extract when concentrated		1.0	Plasticisers + other contaminants *n*-alkanes: traces SFAs: traces
SRP98 Context 15900 – between lead coffin and stone sarcophagus Originally collected for particle size	PSA289	Dark brown soil with inclusions		2.0	Plasticisers + other contaminants *n*-alkanes: traces *n*-alkanols: $C_{24\text{-}30}$ (EOP) SFAs: $C_{16;\ 18}$ – traces; Branched: $C_{14\text{-}16}$ MUFAs: $C_{16:1;\ 18:1}$ Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol

analysis – considered to be soil ingress by excavators **CONTROLS**	PSA291	Dark brown soil with inclusions		2.0	Plasticisers + other contaminants
	PSA292	Dark brown soil with inclusions		2.0	Plasticisers + other contaminants
	PSA293	Dark brown soil with inclusions		2.0	Plasticisers + other contaminants
	PSA294	Dark brown soil with inclusions		2.0	Plasticisers + other contaminants SFAs: C_{10-18} (C_{16} dom); Branched C_{14-16} MUFAs: $C_{16:1;\ 18:1}$

SRP98 Context 15904 – from within lead coffin Originally collected for pollen analysis	P12 <295>	Dark brown soil from gut area		1.0	Plasticisers + other contaminants *n*-alkanols: $C_{14\text{-}30}$ (C_{26} dom); SFAs: $C_{8\text{-}26}$ (EOP, C_{16} dom); Branched: $C_{14;\ 15;\ 16}$; MUFAs: $C_{16:1;\ 18:1}$ Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid *Pistacia* spp.: olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids
	P15 <298>	Dark brown soil from foot of coffin		1.0	Plasticisers + other contaminants *n*-alkanols: $C_{14\text{-}30}$ (EOP; C_{26} dom); SFAs: $C_{8\text{-}28}$ (EOP, C_{16} dom); Branched: $C_{14\text{-}16}$; MUFAs: $C_{16:1;\ 18:1}$; Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid *Pistacia* spp.: olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids

	P16 <329>	Dark brown soil from left side of lower legs		0.6	Plasticisers + other contaminants *n*-alkanols: C_{14-32} (EOP; C_{26} dom); SFAs: C_{8-32} (EOP, C_{16} dom); Branched: $C_{14-17;}$ MUFAs: $C_{16:1;\ 18:1}$; Steroids: cholesterol; β-sitosterol – traces; stigmastanol – traces Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid *Pistacia* spp.: olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids
	P21 <334>	Dark brown soil from right side of right femur		1.0	Plasticisers + other contaminants *n*-alkanols: C_{16-32} (EOP; C_{26} dom); SFAs: C_{8-30} (EOP, C_{16} dom); Branched: $C_{14-17;}$ MUFAs: $C_{16:1;\ 18:1}$; Steroids: cholesterol; β-sitosterol; 5α-cholestanol; stigmastanol Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid *Pistacia* spp.: 28-norolean-12-en-3-one; olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids

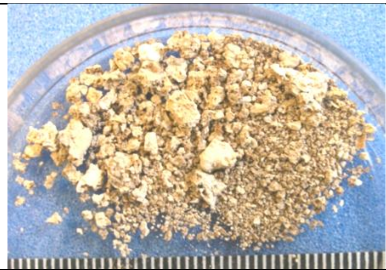
	P22 <335>	Dark brown soil from area of left arm		1.0	Plasticisers + other contaminants 108 BP – ?phenols: 220; 248; 276; 304 Olean-12-en-3-one; 28-norolean-17-en-3-one; oleanonic aldehyde – traces
	P23 <336>	Dark brown soil with pale inclusions from centre of torso		0.6	Plasticisers + other contaminants *n*-alkanols: C_{14-30} (EOP; C_{26} dom); SFAs: C_{8-28} (EOP, C_{16} dom); Branched: $C_{14-17;}$ MUFAs: $C_{16:1; 18:1}$; Steroids; cholesterol; β-sitosterol; 5α-cholestanol Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid – trace levels *Pistacia* spp.: 28-norolean-12-en-3-one; olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids
	P24 <337>	Dark brown soil from area of right arm		0.8	Plasticisers + other contaminants SFAs: $C_{16; 18}$ dehydroabietic acid - trace olean-12-en-3-one; 28-norolean-17-en-3-one; oleanonic aldehyde - traces

	P25 <338>	Dark brown soil with pale inclusions from left of head end of coffin in vicinity of the cranium		0.9	Plasticisers + other contaminants *n*-alkanols: C_{14-30} (EOP; C_{26} dom); SFAs: C_{8-28} (EOP, C_{16} dom); Branched: $C_{14-17;}$ MUFAs: $C_{16:1; 18:1}$; Steroids: cholesterol; 5α-cholestanol Pinaceae: pimaric acid; sandaracopimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid – trace levels *Pistacia* spp.: 28-norolean-12-en-3-one; olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids
	P26 <339>	Dark brown soil with some pale inclusions from right of head end		1.0	Plasticisers + other contaminants *n*-alkanols: C_{14-30} (EOP; C_{26} dom); SFAs: C_{8-30} (EOP, C_{16} dom); Branched: $C_{14-17;}$ MUFAs: $C_{16:1; 18:1}$; Steroids: cholesterol; β-sitosterol; 5α-cholestanol Pinaceae: pimaric acid; *iso*pimaric acid; DDHA; DHA; abietic acid – trace levels *Pistacia* spp.: 28-norolean-12-en-3-one; olean-12-en-3-one; β-amyrin; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanolic acid; oleanonic aldehyde; *iso*masticadienonic acid; masticadienonic acid; 3α-acetoxy-3-epi*iso* + 3-epimasticadienolic acids

6.4 Case Study 4: Lead-lined burials, Winchester, Hampshire (7.5)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

Site/context	GC-MS ref	Description	Image	Mass (g)	Compounds present
St. Martin's Close Winnall, Winchester	F57M	Dark residues on mandible. Resembles a 'tide' mark. Splashes of white material (?plaster)		0.04	Plasticisers + other contaminants *n*-alkanes: $C_{16\text{-}30}$ (C_{18} dom.); phytane SFAs: $C_{12:0\text{-}20:0}$ ($C_{16:0}$ dom.) MUFAs: $C_{18:1}$ Steroids: cholesterol; ?brassicasterol; stigmasterol; β-sitosterol; 5α-cholestanol
	F57S	Dark residues on fragment of scapula. 'Tide' mark effect and splashes of ?plaster.		0.04	Plasticisers + other contaminants *n*-alkanes: $C_{18\text{-}25}$ *n*-alkanols: $C_{17\text{-}25}$ SFAs: $C_{12:0\text{-}20:0}$ Dehydroabietic acid - trace
	F57R	Friable orange fragments in bag of mixed debris (e.g. with snail shells, bird bones, burnt mammal (?human) bone).		0.05	Plasticisers + other contaminants *n*-alkanes: $C_{17\text{-}25}$ *n*-alkanols: $C_{12\text{-}18}$ SFAs: $C_{14:0\text{-}17:0}$
Eagle Hotel Andover Road Winchester Femur	G336F	Darker areas on left femur (and other lower limb bones) and some adhering residue. The 'tide' mark effect was again observed. The presence of agglomerations of clear/white crystals in the cortical bone was also noted.		0.05	Plasticisers + other contaminants *n*-alkanes: $C_{17\text{-}22}$ (C_{18} dom.); phytane *n*-alkanols: $C_{14\text{-}22}$ Steroids: cholesterol

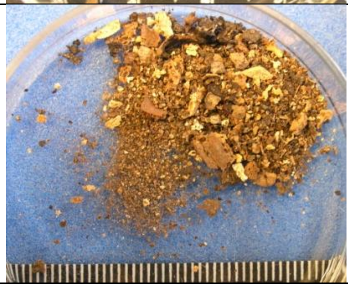
Eagle Hotel Andover Road Winchester 740 <1> **CONTROLS**	WN1 WN7 WN11 WN14 WN16 WN17 WN18	'Contaminated' fill from within the grave cut above the lid of the coffin		2.0 3.0 2.0 2.0 4.0 4.0 4.0	Plasticisers SFAs: $C_{16:0}$; $C_{18:0}$
Eagle Hotel Andover Road Winchester 740<2> **CONTROL**	WN8	Chalk rubble fill within the grave cut adjacent to the coffin		2.0	Traces of phthalate plasticisers
Eagle Hotel Andover Road Winchester 750 <5>	WN2	Mixed material associated with the left radius and ulna		0.5	*n*-alkanes: traces *n*-alkanols: C_{13-30} (EOP), bimodal $C_{18/26}$ SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol methyl dehydroabietate
	WN9	Mixed material associated with the right ribs		1.2	*n*-alkanes: C_{15-29} *n*-alkanols: C_{13-30} (EOP), bimodal $C_{18/26}$ SFAs: $C_{16:0}$ Steroids: cholesterol, campesterol, 5α-cholestanol methyl dehydroabietate

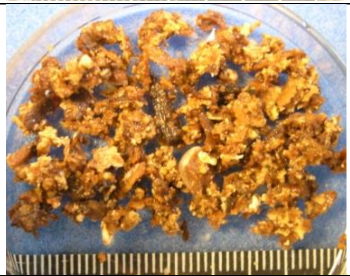
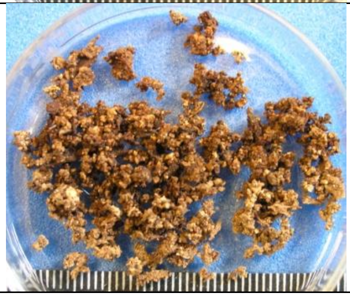
751 <3>	WN4	Light fraction of flot from ‘100% sample from floor of coffin’		0.5	*n*-alkanes: traces
	WN10	1mm fraction from ‘100% sample from floor of coffin’		2.0	*n*-alkanes: C_{15-29} *n*-alkanols: C_{13-30} (EOP), bimodal $C_{18/28}$ SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol, campesterol, β-sitosterol; 5α-cholestanol, campestanol, stigmastanol methyl dehydroabietate
750<5> 751<3>	WN19	Combined sample of material from within coffin		3.0	*n*-alkanes - traces *n*-alkanols: $C_{13-18;\ 24-30}$ SFAs: $C_{14:0;\ 16:0;\ 18:0}$ Steroids: cholesterol; campesterol; stigmasterol; 5α-cholestanol methyl dehydroabietate
751 <4>	WN12	Loose material/‘concreted stalagmite formation’ from the floor of the coffin		2.0 4.0	*n*-alkanols: C_{11-30} (LMM OEP/HMM EOP) SFAs: $C_{12:0-18:0}$, $C_{16:0}$ dom. Steroids: cholesterol, 5α-cholestanol *Pinaceae:* pimaric acid; sandaracopimaric acid; methyl dehydroabietate; dehydroabietic acid

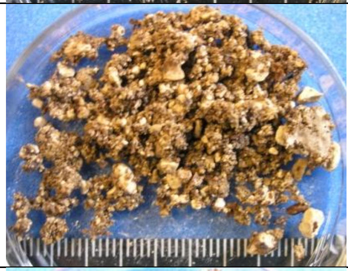
					*Triterpenic compounds:* β-amyrin; α-amyrin; 3α-*epi*-oleanolic; oleanonic acid; oleanolic acid; ursonic acid; ursolic acid
751<3>	WN13	Mineral-replaced textiles and associated material from within the coffin		1.2	*n*-alkanols: $C_{16,\ 18}$ SFAs: $C_{10:0\text{-}20:0}$ (EOP) MUFAs $C_{18:1}$ Steroids: cholesterol *Phenolics*: benzoic acid; vanillin; 3-hydroxybenzoic acid; vanillic acid *Pinaceae traces:* sandarcopimaric acid; isopimaric acid; methyl dehydroabietate; dehydroabietic acid *Triterpenic compounds:* β-amyrin; α-amyrin; 3α-epi-oleanolic acid; oleanonic acid; oleanolic acid; ursonic acid; ursolic acid
750<5> 751<3&4>	WN15	Combined solvent extracts from within coffin	WN9, 10, 12, 13	NA	*n*-alkanes - traces *n*-alkanols: $C_{13\text{-}18}$ SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol; campesterol; stigmasterol; 5α-cholestanol; stigmastanol methyl dehydroabietate *Triterpenic compounds:* β-amyrin; α-amyrin; oleanonic acid

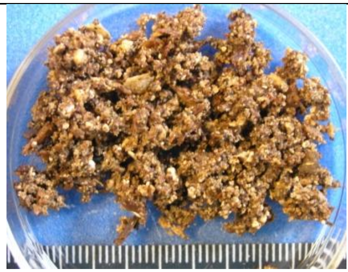
6.5 Case Study 5: Boscombe Down, Wiltshire (7.6)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

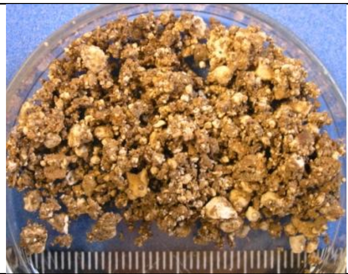
Site Code	Context	Sample Code	GC-MS ref	Description	Image	Mass (g)	Results
56246	SK12787	12787	BD12787	Dry debris associated with adult cranium		1.0	*Plasticisers + other contaminants* ?amines + amides + ?a sulfonamide *n*-alkanes – undifferentiated 'hump' *n*-alkanols: $C_{12\text{-}30}$ (C_{18} dom.) SFAs: $C_{16:0;\ 18:0}$ Sterols: cholesterol Diploptene
	Fill 12824	11387F	BD1	Flot: left side, lower arm, adult		1.0	*Plasticisers + other contaminants* ?amines + amides *n*-alkanes: $C_{15\text{-}17;\ 25\text{-}29}$ - traces *n*-alkanols: $C_{17;\ 24\text{-}30}$ SFAs: $C_{12:0\text{-}26:0}$ ($C_{16:0}$ dom); branched isomers C_{15} + C_{17} UFAs: $C_{18:1;\ 18:2}$ Sterols: cholesterol; cholesta-3,5-dien-7-one Diploptene
	SK12787	11408F	BD2	Flot: left side, foot, adult		0.3	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: $C_{20\text{-}30}$ SFAs: $C_{12:0\text{-}24:0}$ ($C_{16:0}$ dom); branched isomers C_{15}-$_{17}$ MUFAs: $C_{16;1;\ 18:1}$ Steroids: cholesterol, cholesta-3,5-dien-7-one; β-sitosterol; 5α-cholestanol Diploptene + friedelan-3-one

	SK12787	11414F	BD3	Flot: right axial, adult		1.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: $C_{20\text{-}30}$ SFAs: $C_{12:0\text{-}24:0}$ ($C_{16:0}$ dom); branched isomers C_{15}-$_{17}$ MUFAs: $C_{18:1}$ Steroids: cholesterol, cholesta-3,5-dien-7-one; β-sitosterol; 5α-cholestanol Diploptene
	SK12823	11404	BD4	Residue: cranium, child		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: $C_{20\text{-}30}$ SFAs: $C_{14:0;}$ $C_{16:0}$ Steroids: cholesterol, cholesta-3,5-dien-7-one; β-sitosterol Mol. ion 238 BP 167 - unidentified Diploptene
	SK12823	11405	BD5	Residue: axial, child		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene
	SK12823	11406	BD6	Residue: pelvis, child		2.0	Plasticisers + ?other contaminants ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene

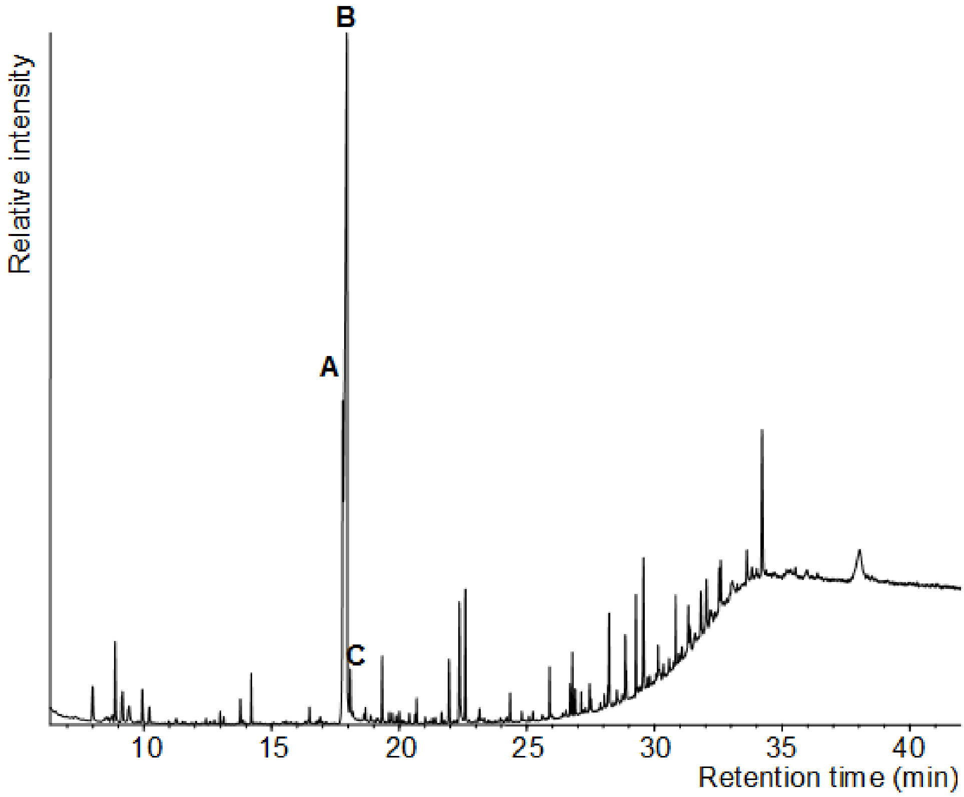
	SK12823	11407	BD7	Residue: knees, child		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol Mol. ion 238 BP 167 - unidentified Diploptene
	Fill 12824	11381	BD8	Residue: left side, feet, adult		2.0	*Plasticisers + other contaminants* ?amines + amides *n*-alkanols: C_{22-30} SFAs: $C_{16:0}$; $C_{18:0}$ Mol. ion 238 BP 167 - unidentified Friedelan-3-one
	Fill 12824	11391	BD9	Residue: right side, ankles, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: C_{20-26} SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol Mol. ion 310 BP 281; Mol. ion 238 BP 167 - unidentified Diploptene + friedelan-3-one
	Fill 12824	11383	BD10	Residue; left side, lower leg, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol Mol. ion 310 BP 281; Mol. ion 238 BP 167 - unidentified Diploptene

	Fill 12824	11393	BD11	Residue: right side, upper leg, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: C_{22-30} SFAs: $C_{14:0-18:0}$ ($C_{16:0}$ dom); Plasticisers + ?other contaminants Mol. ion 310 BP 281; Mol. ion 238 x2 BP 167 + 153; Mol. ion 234 x2 BP 191 + 195 - unidentified Diploptene
	Fill 12824	11385	BD12	Residue; left side, lower pelvis, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes SFAs: $C_{16:0;}$ $C_{18:0}$ Steroids: cholesterol Mol. ion 238 x2 BP 167 + 153 - unidentified Diploptene
	Fill 12824	11395	BD13	Residue; right side, upper pelvis, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes SFAs: $C_{16:0;}$ $C_{18:0}$ Steroids: cholesterol Mol. ion 238 BP 167 - unidentified
	SK12787	11409	BD14	Residue: leg area, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene

	Fill 12824	11387	BD15	Residue; left side, lower arm, adult		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanols: C_{24-28} SFAs: $C_{12:0;}$-$C_{26:0}$; branched isomers C_{15} + C_{17} MUFAs: $C_{18:1}$ Steroids: cholesterol Mol. ion 238 BP 167 - unidentified Diploptene
	Fill 12824	11396	BD16	Right side, adult lower arm		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanes: C_{27-33} Sterols: cholesta-4,6-dien-3-ol; cholesta-3,5-dien-7-one Mol. ion 238 x2 BP 167 + 153 - unidentified Diploptene
	Fill 12824	11397	BD17	Right side, adult shoulder		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene
	SK12787	11412	BD18	Left axial, adult remains		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene

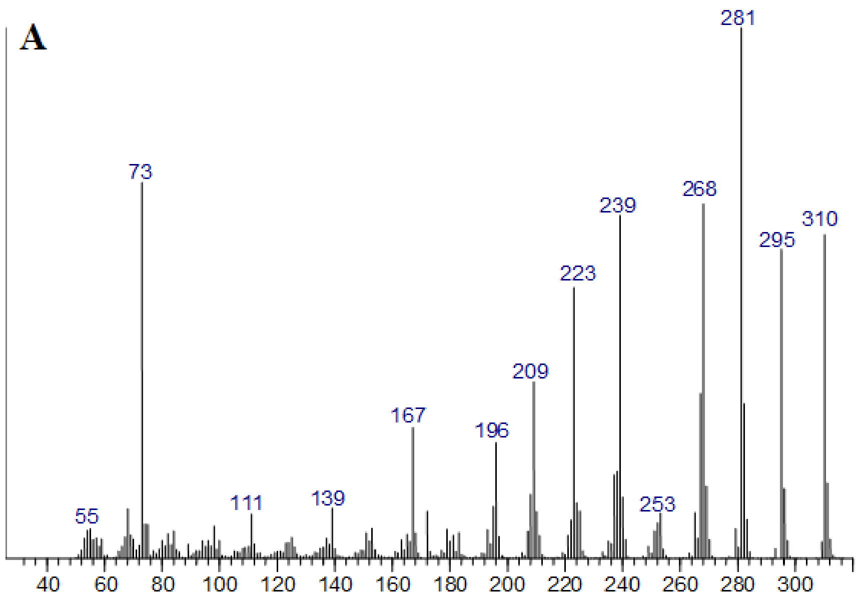
	Fill 12824	11389	BD19	Left side, adult cranium		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes *n*-alkanes: C_{27-33} SFAs: $C_{16:0;}$ $C_{18:0}$ Steroids: cholesterol Mol. ion 310 BP 281 - unidentified Diploptene
	Fill 12824	11398	BD20	Right side, adult cranium		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene
	SK12787	11413	BD21	Area of adult cranium		2.0	*Plasticisers + other contaminants* ?amines + amides + ?aliphatic aldehydes Mol. ion 238 BP 167 - unidentified Diploptene

The three most abundant but unidentified compounds present in many of the samples from Boscombe Down



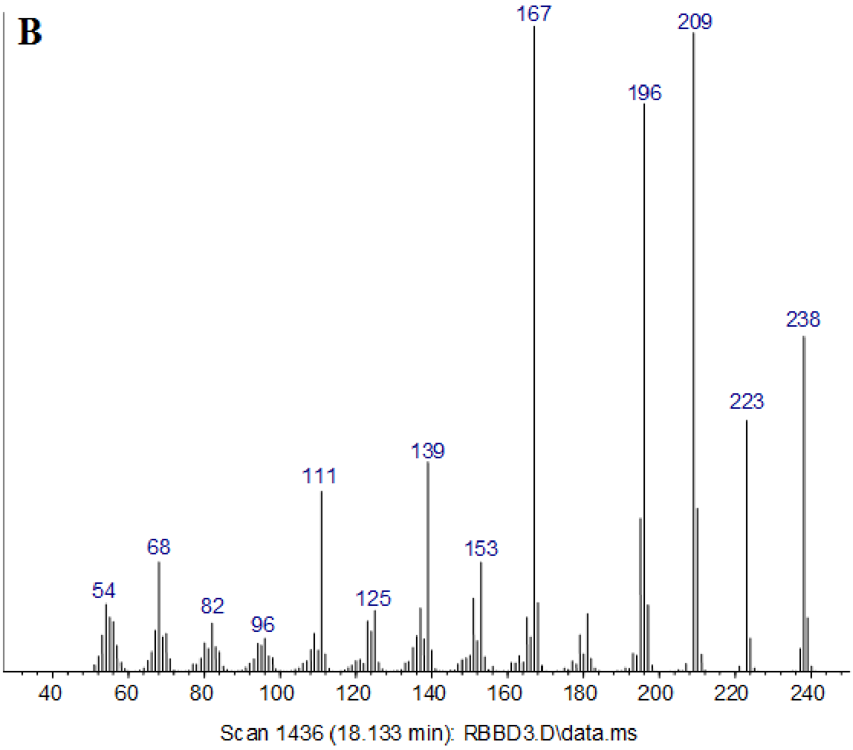
TIC: RBBD2.D\data.ms

	RT	M^+ ion	Fragment ions (BP in bold)
A	17.8	310	295, **281**, 268, 239, 209, 196, 139, 111, 73
B	17.9	238	223, 209, 196, **167**, 153, 139, 111, 68
C	18.1	238	223, 209, 196, 167, **153**, 139, 125, 69

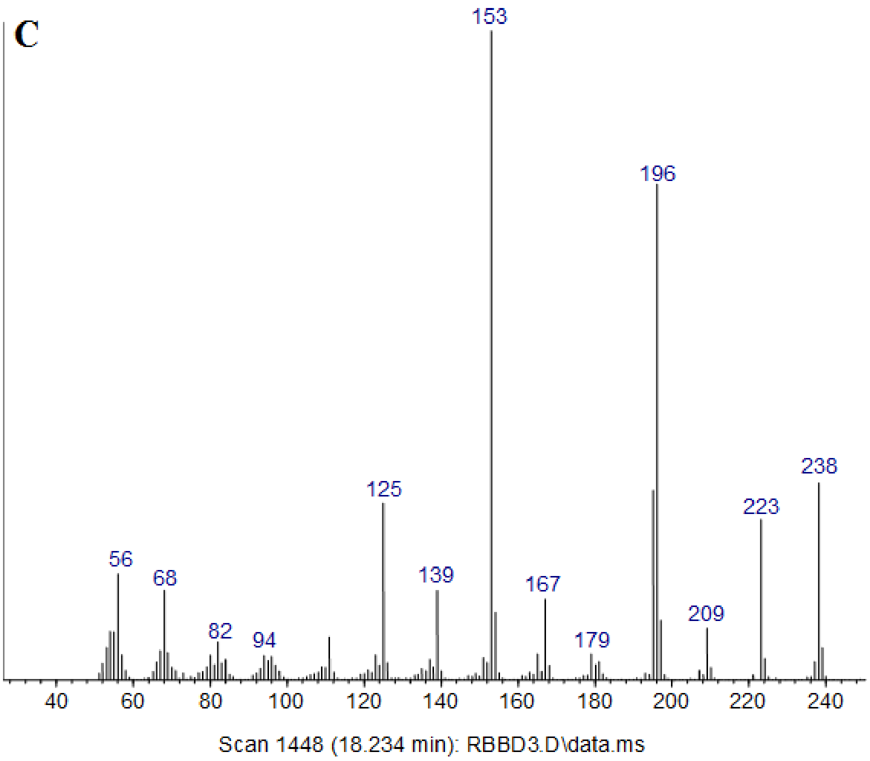


Scan 1395 (17.789 min): RBBD3.D\data.ms

B


167
209
196
238
223
139
111
68
153
54
125
82
96
40
60
80
100
120
140
160
180
200
220
240
Scan 1436 (18.133 min): RBBD3.D\data.ms

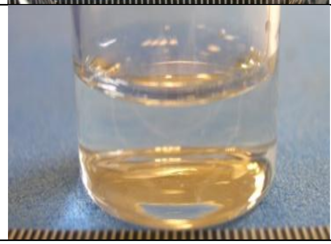
C

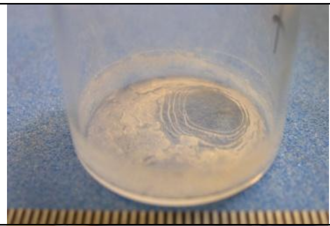

153
196
238
223
125
56
68
139
167
209
82
94
179
40
60
80
100
120
140
160
180
200
220
240
Scan 1448 (18.234 min): RBBD3.D\data.ms

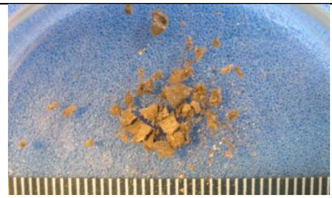
6.6 Case Study 6: Burial ground, Purton, Wiltshire (7.7)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

Site/context	GC-MS ref	Description	Image	Mass (g)	Compounds present
WILT MC890036 Purton 806 Grave 1 Inhumation burial Stone sarcophagus Lead liner	PT1	Detritus associated with the femora. Mixture of materials including bone fragments.		1.5	*Phthalate plasticisers + other contaminants* **SFAs**: $C_{6:0\text{-}20:0}$ (EOP, $C_{16:0}$ max); $C_{14:0\text{-}17:0}$ branched **MUFAs**: $C_{16:1;}$ $C_{18:1}$ isomers **Steroids:** cholesterol; cholestan-3,5-dien-7-one; choles-5-en-3-one; β-sitosterol (trace) **Wax esters**: $C_{40\text{-}46}$ **Diterpenes:** pimaric; isopimaric; didehydroabietic; dehydroabietic; abietic acids **Triterpenes**: 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanonic aldehyde; oleanolic acid; ursolic acid; ?dammaranes (bp 163)
	PT2a	First sample of detritus associated with pieces of lead. Mixture of materials including degraded fragments of lead.		4.0	*Phthalate plasticisers + other contaminants* ***n*-alkanols**: $C_{24\text{-}32}$ (EOP, C_{26} max) **SFAs**: $C_{6:0\text{-}18:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{18:1}$ isomers **Steroids:** cholesterol **Triterpenes**: 28-norolean-12-en-3-one; ?olean-12-en-3-one; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; moronic acid; oleanonic acid; oleanonic aldehyde; oleanolic acid; *iso*masticadienonic acid; masticadienonic acid; ocotillones (bp 143)
	PT2b	Second sample of detritus associated with pieces of lead. Mixture of materials including degraded fragments of lead.		4.0	*Phthalate plasticisers + other contaminants* ***n*-alkanols**: $C_{24\text{-}30}$ (EOP, C_{26} max) **SFAs:** traces **Steroids:** cholesterol; campesterol (trace) **Triterpenes**: 28-norolean-12-en-3-one; ?olean-12-en-3-one; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; oleanonic aldehyde; ?dammaranes (bp 163)

	PT3	Detritus associated with the cranium. Mixture of materials including bone fragments.		1.4	*Phthalate plasticisers + other contaminants* ***n*-alkanols**: C_{24-32} (EOP, C_{26} max) **SFAs**: $C_{8:0-18:0}$ (EOP, $C_{16:0}$ max); $C_{15:0-17:0}$ branched **MUFAs**: $C_{16:1,}$ $C_{18:1}$ isomers **Steroids:** cholesterol **Diterpenes:** pimaric; palustric/levopimaric; isopimaric; didehydroabietic; dehydroabietic; abietic acids **Triterpenes**: 28-norolean-12-en-3-one; ?olean-12-en-3-one; 28-norolean-17-en-3-one; 28-norolean-12,17-dien-3-one; oleanonic aldehyde; ?dammaranes (bp 163)
WILT MC890036 Purton 806 Grave 2 Cremation burial Stone sarcophagus Lead liner Stored in plastic vials.	PT4	Charred bone fragments + associated matter from cremation in glass vessel.		1.0	**SFAs**: $C_{6:0-20:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{18:1}$ isomers **Oxygenated compounds**: C_{8-10} dioic acids; 10-oxooctadecanoic acid; 10-hydroxyoctadecanoic acid **Steroids:** β-sitosterol (trace); stigmastan-3,5-dien-7-one **Terpenoids**: 218 bp; 28-norolean-12-en-3-one; 28-norolean-17-en-3-one; friedelan-3-one; oleanonic acid
	PT5	White residue associated with cremation in glass vessel.		0.3	**SFAs**: $C_{6:0-18:0}$ (EOP, $C_{16:0}$ max) **Terpenoids**: 218 bp; 28-norolean-12-en-3-one; 28-norolean-17-en-3-one; friedelan-3-one; ?oleanonic acid
	PT6	Clear liquid from within glass cremation vessel. No visible residue observed after evaporation.		NA	*Phthalate plasticisers + other contaminants* **No other lipids above limits of detection**

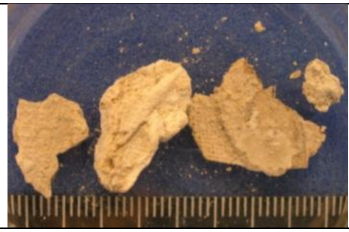
	PT7	Liquid from base of glass cremation vessel. Cream-white residue obtained after evaporation.		NA	*Phthalate plasticisers + other contaminants* **No other lipids above limits of detection**
	PT8	Liquid from top of glass vessel prior to evaporation. Cream-white residue obtained after evaporation.		NA	*Phthalate plasticisers + other contaminants* **No other lipids above limits of detection**
	PT9	Sticky orange-grey matter mixed with bone fragments from within glass vessel.		2.5	**SFAs**: $C_{6:0-18:0}$ (EOP, $C_{16:0}$ max) *TMS hindered* **MUFAs**: $C_{18:1}$ **Steroids:** stigmastan-3,5-dien-7-one (bp 174; $M^{+\cdot}$ 410) **Terpenoids**: 218 bp; 28-norolean-12-en-3-one; 28-norolean-17-en-3-one; friedelan-3-one
	PT10	Cream-grey matter mixed with bone fragments from within glass vessel.		1.2	**SFAs**: $C_{6:0-18:0}$ (EOP, $C_{16:0}$ max) *TMS hindered* **MUFAs**: $C_{18:1}$ **Wax esters**: C_{32-36} **Steroids:** stigmastan-3,5-dien-7-one (bp 174; $M^{+\cdot}$ 410) **Terpenoids**: 218 bp; 28-norolean-12-en-3-one; 28-norolean-17-en-3-one; friedelan-3-one
WILT MC890036 Purton 806 Grave 3 Inhumation burial Normative **CONTROL**	PT11	Soil adhering to the left distal femur and proximal tibia.		0.2	*Phthalate plasticisers + other contaminants* ***n*-alkanes**: C_{17-33} (OEP, bimodal, $C_{19} + {}_{31}$ max)

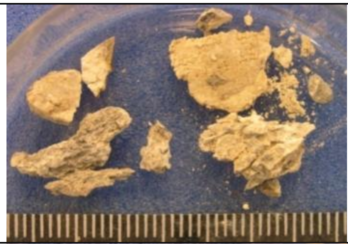
WILT MC890036 Purton 806 Grave 6 Inhumation burial Stone sarcophagus	PT12	Dark residue adhering to the hyoid.		0.1	*Phthalate plasticisers + other contaminants* **SFAs**: $C_{6:0-20:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{16:1;}$ $C_{18:1}$ isomers **Oxidation products:** C_{6-10} dioic acids; 10-oxooctadecanoic acid; 10-hydroxyoctadecanoic acid
WILT MC890036 Purton 806 Grave 6 Inhumation burial Stone sarcophagus	PT13	Dark residue adhering to the cranium. Skeletal remains stored in plastic bags (PT6-2).		0.6	*Phthalate plasticisers + other contaminants* ***n*-alkanes**: C_{17-33} (OEP, bimodal, C_{19} + $_{31}$ max) ***n*-alkanols**: C_{12-32} (EOP, C_{26} max) **SFAs**: $C_{6:0-18:0}$ (EOP, $C_{16:0}$ max) **MUFAs**: $C_{18:1}$ isomers **Steroids:** campesterol (trace) **Hopanes:** 191 bp M+ 384; 412

6.7 Case Study 7: Burial grounds around Dorchester (7.8)

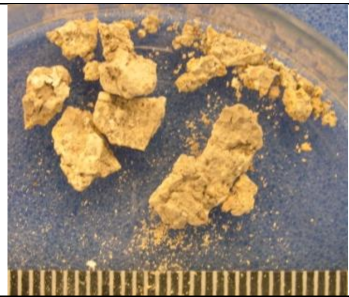
DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

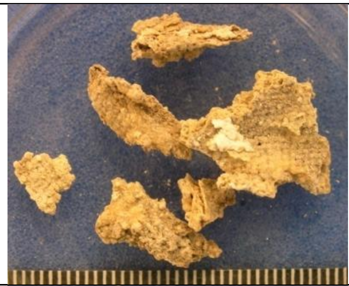
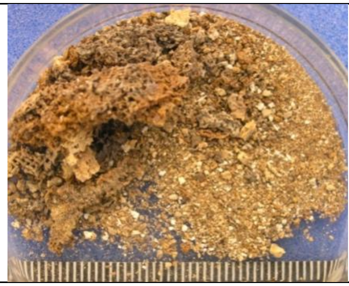
Site/context	GC-MS ref	Description	Image	Mass (g)	GC-MS results
Poundbury R2 mausoleum Grave 8	PD8 G1o **CONTROL**	Residue from outer surface of the 'plaster' (gypsum)		0.6	Traces of *n*-alkanes: C_{16-28}
	PD8 G2i	Residue from inner surface of the 'plaster' (gypsum)		0.4	*n*-alkanes: C_{17-25} *n*-alkanols: C_{12-18} SFAs: $C_{8:0-12:0;\ 14:0;\ 16:0;\ 18:0}$ ($C_{16:0}$ dom) MUFAs: $C_{18:1}$ *Pinaceae:* norabietatraene; ?norabietatriene; simonellite; ?retene pimaric acid; sandaracopimaric acid; isopimaric acid (most abundant); methyl dehydroabietate; DHA acid; abietic acid; 7-oxoDHA acid
	PD8 G3	Darker areas associated with textile impressions on the 'plaster' (gypsum)		0.2	Traces of *n*-alkanes: C_{16-18}

	PD8 G4	Darker areas associated with textile impressions on the 'plaster' (gypsum)		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-26} *Pinaceae*: norabietatetraene; norabietatriene; retene; methyl dehydroabietate
	PD8 G5	Darker areas associated with textile impressions on the 'plaster' (gypsum)		0.8	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-22} *n*-alkanols: C_{12}
Poundbury Site A Grave 49	PD49	Darker areas associated with textile impressions		0.2	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-22}
Poundbury R7 mausoleum Grave 99	PD99a	Coffin debris from around foot		---	Insufficient sample
	PD99b	Orange fragments from below foot		---	Insufficient sample
Poundbury Site E Grave 127	PD127a	Darker areas of residue associated with textile impressions		0.4	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-18} *Pinaceae:* norabietatetraene; norabietatrienes; bis-norabietatriene; methyl dehydroabietate

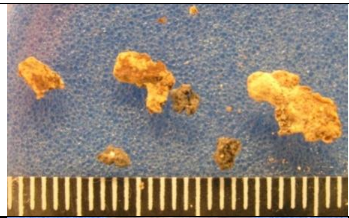
	PD127b	Darker areas of residue		1.0	Traces of *n*-alkanes: C_{18-22}
Poundbury R10 mausoleum Grave 513	PD513	Darker areas associated with textile impressions		0.3	Traces of *n*-alkanes: C_{18-20}
Poundbury R10 mausoleum Grave 517	PD517	Dark residues from voids and areas on the surface of the 'plaster' (gypsum)		0.6	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-26} *n*-alkanols: C_{10-18} SFAs: $C_{8:0-18:0}$ ($C_{9:0}$ dom) *Pinaceae:* norabietatrienes; methyl dehydroabietate
Poundbury R9 mausoleum Grave 529	PD529	Orange residue and adhering fragments near textile impressions on the 'plaster'		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{17-30} *n*-alkanols: C_{10-14} (C_{13} dom) SFAs: $C_{8:0-18:0}$ ($C_{9:0}$ dom) MUFAs: trace of $C_{18:1}$ *Pinaceae:* norabietatetraene; norabietatriene; methyl dehydroabietate

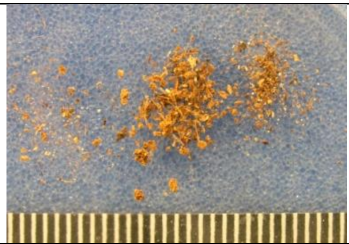
Poundbury R9 mausoleum Grave 530	PD530a	Darker areas associated with textile impressions on the 'plaster' (gypsum)		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-24} *n*-alkanols: C_{10-14} (C_{13} dom) SFAs: $C_{16:0;\ 18:0}$ traces *Pinaceae*: norabietatetraene; norabietatrienes; retene; methyl dehydroabietate
	PD530b	Orange residue near textile impressions on the 'plaster' (gypsum)		0.06	Series: BP 105 Mol. ions 232, 246, 260
	PD530c	Orange-red areas and fragments on the 'plaster' (gypsum)		0.1	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-24} SFAs: $C_{16:0}$ trace
	PD530d	Dark residue and small patches on the 'plaster' (gypsum)		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{15-27} SFAs: $C_{16:0}$; $C_{18:0}$ trace *Pinaceae:* norabietatetraene; norabietatrienes; retene; methyl dehydroabietate

Poundbury Site B Grave 599	PD599	Darker areas associated with textiles impressions		0.2	Below limits of detection
Poundbury Site E Grave 658	PD658	Darker areas associated with textile impressions		0.1	Below limits of detection
Poundbury Site E Grave 775	PD775	Darker areas associated with textile impressions		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-26}
Poundbury Site E Grave 817	PD817	Darker areas associated with textile impressions		1.0	SFAs: $C_{8:0-18:0}$ ($C_{9:0}$ dom) MUFAs: $C_{18:1}$

Poundbury Site E Grave 858	PD858	Darker areas associated with textile impressions		1.0	*n*-alkanes: $C_{16\text{-}26}$ (trace) SFAs: $C_{8:0\text{-}18:0}$ ($C_{9:0}$ dom) MUFAs: $C_{18:1}$
Poundbury Site E Grave 862	PD862	Darker areas associated with textile impressions		0.3	SFAs: $C_{8:0\text{-}18:0}$ ($C_{9:0}$ dom) MUFAs: $C_{18:1}$
Poundbury Site E Grave 867	PD867	Darker areas associated with textile impressions		---	Insufficient sample
Poundbury Site E Grave 868	PD868	Darker areas associated with textile impressions		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: $C_{15\text{-}30}$ *n*-alkanols: $C_{12\text{-}32}$ (EOP; bimodal $C_{12/28}$) SFAs: $C_{8:0\text{-}18:0}$ ($C_{16:0}$ dom) MUFAs: $C_{18:1}$ Steroids: cholesterol

Poundbury Site E Grave 892	PD892	Darker areas associated with textile impressions		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{14-28} *n*-alkanols: C_{12-32} (EOP bimodal $C_{12/28}$) SFAs: $C_{8:0-30:0}$ ($C_{16:0}$ dom) MUFAs: $C_{18:1}$ Steroids: cholesterol; 5α-cholestanol; β-sitosterol *Pinaceae*: norabitatetraene; norabietatriene; methyl dehydroabietate; DHA acid; 7-oxoDHA acid
Poundbury Site E Grave 922	PD922	Darker areas associated with textile impressions		---	Insufficient sample
Poundbury Site E Grave 1040	PD1040	Darker areas associated with textile impressions		1.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-29} *n*-alkanols: C_{12-28} (EOP bimodal $C_{12/28}$) SFAs: $C_{8:0-18:0}$ ($C_{16:0}$ dom) MUFAs: $C_{18:1}$ Steroids: cholesterol *Pinaceae*: retene; methyl dehydroabietate; DHA acid; 7-oxoDHA acid
Crown Buildings Plaster burial in lead-liner	CGB H1	Fragments of hair. Solvent washed in DCM.		NA	SFAs: $C_{16:0;\ 18:0}$ MUFAs: $C_{18:1}$ trace
	CGB H2	End of plait. Solvent washed in DCM.		NA	SFAs: $C_{16:0}$; $C_{18:0}$ trace

	CGB V	Associated material ?organic debris		0.05	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{16-24} *n*-alkanols: C_{12-16} traces SFAs: $C_{8:0-18:0}$ ($C_{16:0}$ dom)
Alington Ave Grave 3664	AA3664C	Sub-sample of debris associated with the cranium		2.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{15-29} (C_{17} dom) *n*-alkanols: C_{20-30} (evens; C_{28} dom) SFAs: $C_{14:0;\ 16:0;\ 18:0}$ *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives α- and β-boswellic acids
	AA3664 F	Residue adhering to foot bone		---	Insufficient sample
Alington Ave Grave 4378	AA1 C1	Sub-sample of debris from around the head end of the coffin		2.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{15-29} (C_{17} dom) *n*-alkanols: C_{20-30} (EOP; C_{28} dom) SFAs: $C_{14:0;\ 16:0;18:0}$ *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives α- and β-boswellic acids
	AA6 C2	Second sub-sample of debris from around the head end of the coffin as confirmation		2.0	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: C_{15-29} (C_{17} dom) *n*-alkanols: C_{20-30} (EOP; C_{28} dom) SFAs: $C_{14:0;\ 16:0;18:0}$ *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives α- and β-boswellic acids

	AA3 M	Dark areas of residue from mandible		0.05	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: traces *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives α- and β-boswellic acids - traces
	AA4 R	Dark area of residue from rib bones		0.05	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: traces *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives
	AA5 CV	Debris associated with cervical vertebrae		0.1	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: traces *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives
	AA7 H	Dark areas of residue from humerus and associated debris		0.5	Series: BP 105 Mol. ions 232, 246, 260 *n*-alkanes: $C_{14\text{-}29}$ (C_{17} dom) *n*-alkanols: $C_{20\text{-}30}$ (EOP; C_{28} dom) SFAs: $C_{14;0;\ 16:0;\ 18:0}$ *Boswellia* spp.: noroleanene and norursene compounds α- and β-amyrin and derivatives α- and β-boswellic acids

6.8 Case Study 8: Recent find near Ilchester, Somerset (7.9)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

All of the samples appeared to comprise soil ingress which completely filled the lead-liner, encasing the skeleton.

Sample code (grid number)	GC-MS ref	Sample location	Mass (g)	Compounds present
A104 **CONTROL**	SSCN	Above the skeleton	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{14:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol **Terpenes:** β + α-amyrin
B201	SS1	Left of the cranium, central in coffin	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-34} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{14:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol **Terpenes:** β + α-amyrin
B302	SS2	Left of mandible, below bent over side of lead-liner	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{18-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs**: 1-monopentadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol

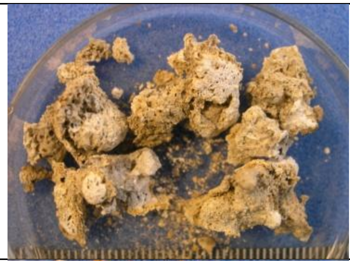
B103	SS3	Right proximal humerus	3.0	***n*-alkanes**: C_{19-35} (OEP, C_{31} max) ***n*-alkanols**: C_{16-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs**: 1-monopentadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B203	SS4	Area of the ribs and vertebrae, central in coffin	3.0	***n*-alkanes**: C_{19-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs**: 1-monopentadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B304	SS5	Left proximal ulna + radius, below bent over side of lead-liner	3.0	***n*-alkanes**: C_{19-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-36} (EOP, C_{26} max) ***n*-alkanones + glycerols**: traces **SFAs**: $C_{12:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B105	SS6	Right lower arm, right edge of coffin	3.0	***n*-alkanes**: C_{21-35} (EOP, C_{31} max) ***n*-alkanols**: C_{18-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs:** 1-monopenta/hexadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B205	SS7	Gut area/left os coxa, central in coffin	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{18-34} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-18:0}$ (EOP, $C_{16:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs:** 1-monopenta/hexadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol **Terpenes:** β + α-amyrin
B306	SS8	Left hand bones, left edge of coffin, below bent over side of lead-liner	3.0	***n*-alkanes**: C_{19-35} (OEP, C_{31} max) ***n*-alkanols**: C_{18-36} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{12:0-32:0}$ (EOP, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs:** 1-monopenta + hexadecanoin + other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B107	SS9	Gap between right femur and tibia, right side of coffin	3.0	***n*-alkanes**: C_{21-35} (OEP, C_{31} max) ***n*-alkanols**: C_{18-36} (EOP, C_{26} max) ***n*-alkanones**: traces

				SFAs: $C_{12:0-30:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs:** 1-monopenta + hexadecanoin + other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B207	SS10	Near hobnails in gap between left femur and tibia, central in coffin	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{18-34} (EOP, C_{26} max) ***n*-alkanones**: traces **SFAs**: $C_{14:0-30:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **MAGs:** 1-monopenta/hexa/heptadecanoin; other glycerols **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B308	SS11	Empty area, distal third, left side of coffin, below bent over lead-liner	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-34} (EOP, C_{26} max) ***n*-alkanones + glycerols**: traces **SFAs**: $C_{14:0-28:0}$ (EOP, bimodal, $C_{16:0/26:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: cholesterol; cholestanol; campesterol; stigmasterol; β-sitosterol; stigmastanol
B109	SS12	Area of foot bones, left distal corner of the lead-liner	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-34} (EOP, C_{26} max) ***n*-alkanones + glycerols**: traces **SFAs**: $C_{14:0-18:0}$ (EOP, $C_{16:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: traces
B209	SS13	Area of hobnails, centre of distal edge of the lead-liner	3.0	***n*-alkanes**: C_{21-33} (OEP, C_{31} max) ***n*-alkanols**: C_{20-34} (EOP, C_{26} max) ***n*-alkanones + glycerols**: traces **SFAs**: $C_{14:0-18:0}$ (EOP, $C_{16:0}$ max); **br:** C_{15-17} ; **MUFAs**: $C_{16:1}$; $C_{18:1}$ **Sterols**: traces

6.9 Case Study 9: Plaster burials around York (7.10)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient.

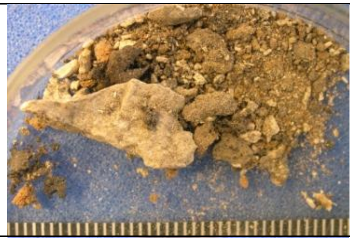
Site/context	GC-MS ref	Description	Image	Mass (g)	Compounds present
YORYM: 2007.6207 Plaster burial Full body cast	YK1	Cranial region Orange fragments and dark staining on plaster associated with mineral replaced textiles		1.5	Plasticisers and other contaminants
	YK2	Leg region Orange fragments and dark staining on plaster associated with mineral replaced textiles		2.0	Plasticisers and other contaminants
YORYM: 2007.6206 Railway excavations Cist tomb Cedar wood coffin Plaster burial Encased in bubble wrap.	YK3	Cranial region Dark staining on plaster associated with mineral replaced textiles.		4.0	*n*-alkanes - traces SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol *Triterpenic compounds:* series of olean-12-ene and urs-12-ene derivatives base peak *m/z* 218
	YK4	Cranial region Yellow-brown staining on plaster associated with mineral replaced textiles.		2.0	*Sesquiterpenes:* curcumene; cuparene; longipinene; calacorene; longifolene; calamenene; related compounds

	YK5	Cranial region Yellow-brown stained mass of mineral replaced textiles.		2.0	SFAs*:* $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol, campesterol *Sesquiterpenes* - traces *Triterpenic compounds:* series of olean-12-ene and urs-12-ene derivatives base peak *m/z* 218
YORYM: 2007.6208 Plaster burial	YK6	Leg region Orange residue embedded in plaster associated with mineral replaced textiles		0.3	Plasticisers and other contaminants
	YK7	Leg region Dark staining on plaster associated with mineral replaced textiles		1.0	Plasticisers and other contaminants
YORYM: 2007.6214 Plaster burial	YK8	Leg region Dark staining on plaster associated with mineral replaced textiles		0.6	*n*-alkanes - traces *n*-alkanols - traces SFAs*:* $C_{16:0}$; $C_{18:0}$ Wax esters*:* C_{36-42} *PAHs:* phenanthrene; anthracene; fluoranthrene; pyrene; benzoanthracene; isochrysene; chrysene; indenopyrene; benzoperylene *Hopanes*: C_{27-31} isomers *Triterpenic compounds:* β- and α-amyrin and related compounds

YORYM: 2007.6205i Mill Mount Plaster burial	YK9	Cranial region Dark staining on plaster associated with mineral replaced textiles		2.0	*n*-alkanes - traces, bimodal *Triterpenic compounds:* series of olean-12-ene and urs-12-ene derivatives base peak *m/z* 218
	YK10	From gap in plaster - soil sample **CONTROL**		0.5	*n*-alkanes *n*-alkanols: C_{24-30} SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: β-sitosterol
YORYM: 2013.152 Stone sarcophagus Plaster burial Infant	YK11	Cranial region Dark staining and orange fragments on plaster associated with mineral replaced textiles		2.0	Plasticisers and other contaminants
YORYM: 2010.1201 Railway excavations Inscribed sarcophagus Plaster burial Hobnails + iron studs Some skeletal remains	YK12	Residue from base of sarcophagus		4.0	*Hopanes*: C_{27-31} isomers *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin)

	YK13	Residue from base of sarcophagus		4.0	*Hopanes*: $C_{27\text{-}31}$ isomers *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin)
	YK14	Residue from base, fine silt remaining in base of sarcophagus		4.0	*n*-alkanes (bimodal) *n*-alkanols: $C_{24\text{-}30}$ SFAs: $C_{16:0}$; $C_{18:0}$ Steroids: cholesterol, campesterol, β-sitosterol, stigmasta-4-en-3-one Wax esters: $C_{38\text{-}42}$ *Hopanes*: $C_{27\text{-}31}$ isomers *Triterpenic:*β- + α-amyrin + related compounds *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin)
YORYM: 2007.6212 Railway excavations Lead coffin Plaster burial Child 3rd-4th c. AD	YK15	'Linen cast' Yellow stained linen fragments adhering to plaster		0.5	*n*-alkanes
YORYM: 2007.6213 Plaster burial	YK16	Yellow/orange staining on plaster associated with mineral replaced textiles		0.7	*n*-alkanes Steroids: cholesterol *Hopanes*: $C_{27\text{-}31}$ isomers *Triterpenic compounds:* nor-olean-17-en-3-one and related compounds

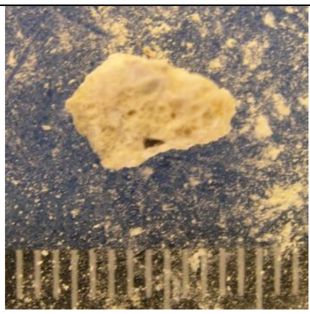
	YK17	Dark residue External surface		4.0	*n*-alkanes *Hopanes*: C_{27-31} isomers *Triterpenic compounds:* nor-olean-17-en-3-one and related compounds
YORYM: 2007.6211 Heslington Field Stone sarcophagus Plaster burial Adult female 4th c. AD	YK18	Black/orange fragments adhering to plaster associated with mineral replaced textiles		0.7	Below levels of detection
YORYM: 2007.6209 89 The Mount Plaster burial	YK19	?Soil adhering to plaster next to portion of humerus		4.0	*n*-alkanes - traces *n*-alkanols: C_{22-28}
YORYM: 2007.6209 89 The Mount Plaster burial	YK20	Soil ingress associated with plaster **CONTROL**		4.0	*n*-alkanes *n*-alkanols: C_{26}

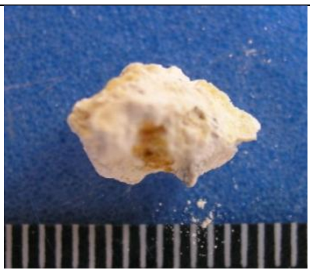
YORYM: 2007.6210 Lead-lined coffin Plaster burial	YK21	Minimal residues, very hard surface to plaster and grey-discoloration due to lead ion substitution?		0.8	*n*-alkanes Steroids: cholesterol, campestanol, β-sitosterol, stigmastanol + derivatives *Hopanes*: $C_{27\text{-}31}$ isomers
YORYM: 2010.1196 Stone sarcophagus Lead-liner Plaster burial Late 2^{nd}-4^{th} C. AD	YK22	Debris from base of lead liner		4.0	*n*-alkanes ($C_{17\text{-}39}$) + pristane + phytane Steroids: cholesterol, campesterol, stigmasterol, β-sitosterol *PAHs*: phenanthrene, fluoranthene, pyrene, benzoanthracene, chrysene *Steranes: $C_{27\text{-}31}$ ααβ/αββ* *Hopanes*: $C_{27\text{-}33}$ isomers *Triterpenic:*β- + α-amyrin + related compounds *Contaminants:* chlorinated + brominated compounds (e.g. dieldrin, endrin, ?benzene hexachloride)
	YK23	Residues from the plaster		0.4	*n*-alkanes *Hopanes*: $C_{27\text{-}33}$ isomers *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin, ?benzene hexachloride)
YORYM: 1980.51 Catterick Plaster burial	YK24	Cranial region External surface Soil residue **CONTROL**		2.0	*n*-alkanes

YORYM: 2010.1219 Railway excavations Stone sarcophagus Plaster burial Modified for display under glass	YK25	Debris from foot end of base of sarcophagus		4.0	*Triterpenic compounds:* oleanonic acid, oleanolic acid + derivatives *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin, ?benzene hexachloride)
	YK26	Debris from mid-region of base of sarcophagus, left wrist and hand bones still in situ		4.0	*n*-alkanols: C_{24-30} SFAs: $C_{16:0;}$ $C_{18:0}$ MUFAs: $C_{18:1}$ x2 isomers Steroids: cholesterol, campesterol, stigmasterol, β-sitosterol *Triterpenic compounds:* oleanonic acid, oleanolic acid and derivatives *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin, ?benzene hexachloride)
	YK27	Debris from head end of base of sarcophagus		4.0	*Triterpenic:* oleanonic acid, oleanolic acid + derivatives *Contaminants*: chlorinated + brominated compounds (e.g. dieldrin, endrin, ?benzene hexachloride)
YORYM: 2007.6126 Bishopsgate Street Clementhorpe Stone sarcophagus Lead-liner Plaster burial Infant + adult female ***or*** x3 individuals	YK28	Minimal residues, very hard surface to plaster, grey-discoloration due to lead ion substitution?		0.06	*n*-alkanes
YORYM: ??? Railway Station , Lead-liner + lid. Plaster burial. Textile fragments. Child c. 7 years old					No visible residues
YORYM: 1971.303, Trentholme Drive, Stone sarcophagus, Grave 196, Male, sub-adult					No visible residues

6.10 Case Study 10: Mersea Island barrow, Essex (7.11)

DCM:MeOH (2:1, v/v), silylation (BSTFA + 1% TMCS, ~0.05 ml). Agilent 7890A GC with 5975C inert XL triple axis MS, column 30 m x 0.25 mm, 0.25 µm HP-5MS), 50°C (isothermal, 2 min) to 350°C (isothermal, 10 min),10°C per min gradient

GC-MS ref	Mass (g)	Description	Image	Compounds present
MS1	0.2	White amorphous mass		*Carboxylic acids:* SFAs $C_{16:0}$; $C_{18:0}$ MUFAs $C_{18:1}$ *Monoterpenes* α-pinene (1R + 1S) β-pinene α-phellandrene camphene menthene o-cymene + β-terpinyl acetate β-phellandrene δ-3-carene γ-terpinene cymenene + terpinolene ocimene dihydrocarvone pinocarvone isocineole (1,4-cineole) α-terpineol verbenone (2-pinen-4-one) *Sesquiterpenes* α-copaene β-elemene β-caryophyllene α-caryophyllene aromadendrene γ-muurolene eudesma-4(14),11-diene α-muurolene + α-selinene γ-cadinene δ-cadinene calamenene α-calacorene carophyllene oxide ?longifolene τ-muurolene *Diterpenoids* (Pinaceae): neutral abietane derivatives *Diterpenoids (Boswellia* spp.): *m*-camphorene; *p*-camphorene; cembrene C; verticilla-4(20),7,11-triene; incensol (underivatised + TMS); incensol oxide *Triterpenic compounds (Boswellia* spp.): 24-norolenan-3,9(11),12-triene; 24-norursa-3,9(11),12-triene; 24-norolean-3,12-diene; 24-norursa-3,12-diene; 3-epi-β-amyrin; 3-epi-α-amyrin; 24-norursa-3,12-diene-11-one; β-amyrenone; 3β-hydroxy-olean-12-en-3-ol (β-amyrin); α-amyrenone; 3β-hydroxy-urs-12-en-3-ol (α-amyrin); 3-hydroxyolean-12-en-23-oic acid (α-boswellic acid); 3-hydroxyurs-12-en-23-oic acid (β-boswellic acid)

MS2	0.1	Inner portion with dark orange inclusions		*Carboxylic acids:* SFAs $C_{16:0}$; $C_{18:0}$ MUFAs $C_{18:1}$ *Monoterpenes and sesquiterpenes - as above* *Diterpenoids* (Pinaceae): neutral abietane derivatives; pimaric acid; sandaracopimaric acid; *iso*pimaric acid; didehydroabietic acid; dehydroabietic acid; abietic acid *Diterpenoids* (*Boswellia* spp.): cembrene C; verticilla-4(20),7,11-triene incensol (underivatised + TMS); incensol oxide *Triterpenoids* (Burseraceae) - *as above*
MS3	0.1	White material that had become powdered		*As above - MS2*

Appendix 7. Dissemination of research

7.1 Reports for the contributing museums

- *Report on the resinous material found with a Roman period infant inhumation from Arrington, Cambridgeshire* (2012) for the Museum of Anthropology and Archaeology, Cambridge; Cambridgeshire
- *Report on the organic residues from late Roman package burials, Dorchester* (2012) for the Dorset County Museum/Dorset Natural History and Archaeology Society, Dorchester, Dorset
- *Report on the organic residues from the late Roman burial known as the 'Spitalfields Lady' (SK15903, SRP 98)* (2012) for the Centre for Human Bioarchaeology (CHB), Museum of London, London
- *Report on the organic residues from late Roman package burials, Winchester* (2012) for Winchester Museums, Hampshire;
- *Report on organic residues from late Roman inhumation burials in the cemeteries around London* (2013) for the CHB, Museum of London, London
- *Report on the organic residues from the late Roman sarcophagus burial from Boscombe Down, Wiltshire, UK* (2013) for Wessex Archaeology, Salisbury, Wiltshire
- *Analysis of organic matter from a cremation urn, Mersea Island* (2013) for J. McKinley, Wessex Archaeology, Salisbury, Wiltshire/Mersea Museum Trust, Mersea Island, Essex
- *Report on the organic residues from the late Roman lead-lined coffin burial (G336), Eagle Hotel site, Winchester, UK* (2013) for Winchester Museums, Hampshire
- *Supplementary report - more residues from late Roman inhumation burials in the cemeteries around London* (2014) for the CHB, Museum of London, London
- *Organic residue analysis of soil samples from the 'Lady in Lead', Somerset* (2014) for Robert Croft, County Archaeologist, Somerset County Council, Somerset
- *Pilot study: organic residue analysis of materials detached from Ancient Egyptian mummies* (2014) for Lidija McKnight, Ancient Egyptian Animal Bio-Bank, University of Manchester, Manchester
- *Organic residue analysis of samples from F77, Bezannes* (2014) for Denis Bouquin, ReimsMétropole, Reims, France
- *Pilot study: molecular analysis of materials used in the embalming of Egyptian avian votive mummies* (2014) for Lidija McKnight, Ancient Egyptian Animal Bio-Bank, University of Manchester, Manchester
- *Report on the analysis of organic residues from Roman period plaster burials around York, North Yorkshire, UK* (2014) for York Museums Trust, York, North Yorkshire

- *Report on the analysis of organic residues from Roman period burials, Northview Hospital, Purton, Wiltshire, UK* (2014) for Swindon Museum and Art Gallery, Swindon, Wiltshire

7.2 Podium presentations

- *The 'semblance of immortality'? molecular identification of resins in mortuary contexts from Roman Britain and evaluation of their significance* (2012), presentation, Royal Society of Chemistry Postgraduate Symposium, University of Bradford, Bradford, UK
- *The 'semblance of immortality'? molecular identification of resins in mortuary contexts from Roman Britain and evaluation of their significance* (2012) research colloquium given to the Department of Conservation and Scientific Research, British Museum, London, UK
- *The 'semblance of immortality'? Resinous materials and late Roman mortuary rites in Britain* (2013), presentation, Experimental and Environmental Archaeology, UK Archaeological Science conference, University of Cardiff, Cardiff, UK
- *'Embalming' in late Roman Britain: resinous materials and their significance in mortuary rites* (2013) presentation, 15th annual conference of the British Association for Biological Anthropology, University of York, York, UK (winner of podium presentation prize)
- *'In a molecular fashion': the identification of resinous materials in Roman mortuary contexts* (2013) presentation (given by invitation) research seminar, 'Matériaux minéraux et organiques en context funéraire' for La Fédération des Sciences Archéologiques de Bordeaux, Université Bordeaux 1, Bordeaux, France
- *The 'semblance of immortality'? resinous materials and mortuary rites in Roman Britain* (2013), research seminar, Archaeology Guest Lectures, University of Bradford, Bradford, UK
- *'Choicest unguents': molecular characterisation of resinous materials from Roman mortuary contexts* (2014) presentation, InterArChive; expanding the horizons of human burial research symposium, University of York, York, UK
- *Mass spectral elucidation of triterpenoid biomarkers of archaeological interest and the validity of APCI for distinguishing between their epimers* (2014), joint presentation with Chloe Townley, Royal Society of Chemistry Postgraduate Symposium, University of Bradford, Bradford, UK
- *'Choicest unguents': molecular evidence for resinous materials from mortuary contexts in Roman Britain* (2014) presentation, 20th annual meeting, European Association of Archaeologists, Istanbul, Turkey
- *'Choicest unguents': resins and mortuary rites in Roman Britain* (2014), presentation (by invitation), Science in Archaeology, annual conference, The Royal Archaeological Institute, University of Bradford, Bradford, UK

- *'The final masquerade'? Resinous substances and mortuary rites in Roman Britain* (2015), presentation (by invitation) Theoretical Roman Archaeology Conference (#TRAC25), School of Archaeology and History, University of Leicester, Leicester, UK
- *'Unparalleled opportunities': molecular analysis of Egyptian votive mummies* (2015), Experimental and Environmental Archaeology, UK Archaeological Science conference, University of Durham, Durham, UK

7.3 Posters

- *Mass spectral elucidation of triterpenoid biomarkers of archaeological interest and the validity of APCI for distinguishing between their epimers* (2014), annual meeting, British Mass Spectrometry Society, AstraZeneca, Alderley Park, Cheshire, UK
- *Mass spectral elucidation of triterpenoid biomarkers of archaeological interest and the validity of APCI for distinguishing between their epimers* (2014), School of Life Sciences, Research and Development Open Day, University of Bradford, Bradford, UK;
- *Choicest unguents': resinous materials and mortuary contexts in Roman Britain* (2014), annual meeting, Anatomical Society, University of Bradford, Bradford, UK
- *Assignment of the 3-OH epimers of the pentacyclic triterpenoids oleanolic and ursolic acid by positive mode APCI* (2015), Royal Society of Chemistry conference, University of Bradford, Bradford, UK

7.4 Outreach

- Presentations given to final year undergraduates and masters students studying for Archaeological Sciences degrees (2012-2014)
- Talks about the research given to members of the general public on Researchers Night, York Museum, York, UK (2014)
- *'Choicest unguents': resins and mortuary rites in Roman Britain*, two presentations (given by invitation) to members of Essex Society for Archaeology and History (ESAH) and supporters of the Mersea Museum Trust as part of a programme of events, ESAH visit to West Mersea, Mersea Island, Essex, UK (2015)
- Posters of results and information provided to Lidija McKnight to form part of the touring exhibition *'Gifts for the Gods: Animal Mummies Revealed'* (2015-2016).

7.5 Publications (in date order)

- Brettell, R., Stern, B., Reifarth, N. and Heron, C. (2014) The '*semblance of immortality'*? Resinous materials and mortuary rites in Roman Britain. *Archaeometry* 56: 444-459.

- Schotsmans, E.M.J., Wilson, A.S., Brettell, R., Munshi, T. and Edwards, H.G.M. (2014) Raman spectroscopy as a non-destructive screening technique for studying white substances from archaeological and forensic burial contexts. *Journal of Raman Spectroscopy* 45: 1301-1308.
- Brettell, R., Schotsmans, E.M.J., Walton Rogers, P., Reifarth, N., Redfern, R.C., Stern, B. and Heron, C.P. (2015a) '*Choicest unguents*: molecular evidence for the use of resinous plant exudates in late Roman mortuary rites in Britain. *Journal of Archaeological Science* 53: 639-648.
- Brettell, R., Stern, B., Heron, C.P. (2015b) Mersea Island barrow: molecular evidence for frankincense. *Essex Society for Archaeology and History* 4 (2013): 81-87.
- Brettell, R., Martin, W., Atherton-Woolham, S., Stern, B. and McKnight, L. (2015c) Organic residue analysis of Egyptian votive mummies and their research potential. *Studies in Conservation* http://dx.doi.org/10.1179/2047058415Y.0000000027.
- Townley, C., Brettell, R.C., Bowen, R.D., Gallagher, R.T., Martin, W.H.C. (2015) The application of positive mode atmospheric chemical ionisation to distinguish epimeric oleanolic and ursolic acids. *European Journal of Mass Spectrometry* 21: 433-442.
- Brettell, R. (2015) Molecular characterisation of natural products. In McKnight, L.M. and Atherton-Woolham, S.D. (authors/editors) *Gifts for the Gods: Ancient Egyptian animal mummies and the British*. Liverpool: Liverpool University Press. 90-91.
- Brettell, R. and Heron, C. (2015) The fragrant dead: how to treat the departed, Roman style. *Current Archaeology* 312: 34-39.
- Brettell, R. (2016) Organic residues from Roman mortuary contexts. In Historic England *Organic residue analysis and archaeology: guidance for good practice document*. Swindon: Historic England. case study.
- Brettell, R., Schotsmans, E., Martin, W., Stern, B. and Heron, C. (with editor) The final masquerade: resinous substances and Roman mortuary rites. In Livarda, A., Riera Mora, S. and Madgwick, R. (editors) *The bioarchaeology of ritual and religion*: Oxford: Oxbow Books. xxxx.
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7.6 Other projects initiated by aspects of the research (in date order)

- Harrison, W., Brettell, R. and Stern, B. (2013) *A multi-technique approach to archaeological soil analysis.* Report prepared for the Royal Society of Chemistry. Bradford, UK: The authors c/o Archaeological Sciences, University of Bradford
- Gamon, A. (2014) *Development and application of analytical methods for the analysis of different natural dyes including, Tyrian purple, indigo, red, blue and black inks components.* M.Sc. Dissertation, University of Bradford, UK
- Harrison, W. (2014) *A multi-technique analysis of Roman era grave soil deposits*. B.Sc. Dissertation. University of Bradford, UK

- Harrison, W.J. (2015) *The characterisation of plant gum sugars and their identification in Ancient Egyptian votive mummies.* M.Sc. Dissertation. University of Bradford, UK.
- Hopkinson, S. (2014) *"The Lady in Lead": an osteo-biography of a Roman individual found in a lead coffin.* M.Sc. Dissertation, University of Bradford, UK
- Townley, C. (2014) *The application of positive mode atmospheric chemical ionisation to distinguish epimeric oleanolic and ursolic acids.* Research undertaken as part of a BMSS Summer Research Scholarship, Chemistry and Forensic Sciences, University of Bradford, UK

Disc 1 (see Appendices 2.3-2.4, 4, 5.1-5.2, 6.1-6.10 for details of contents)